JOURNAL OF

METALS

MAY 1958

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JOURNAL OF

METALS

VOL. 10, NO. 5 MAY 1958

Published to provide a continuing, authoritative, and up-to-date record of technological, engineering, and economic progress in all branches of the industry by The Metallurgical Society, American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc., 29 West 39th Street, New York 18, N. Y.

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COVER

For May, representative forms and shapes of the nuclear age caught the eye of Cover Designer, Naomi Lazard. Although fuel elements and spherical reactor housings dominate the cover, this issue is devoted to the whole range of metals that have received impetus in this nuclear age.

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(Continued on page 314)

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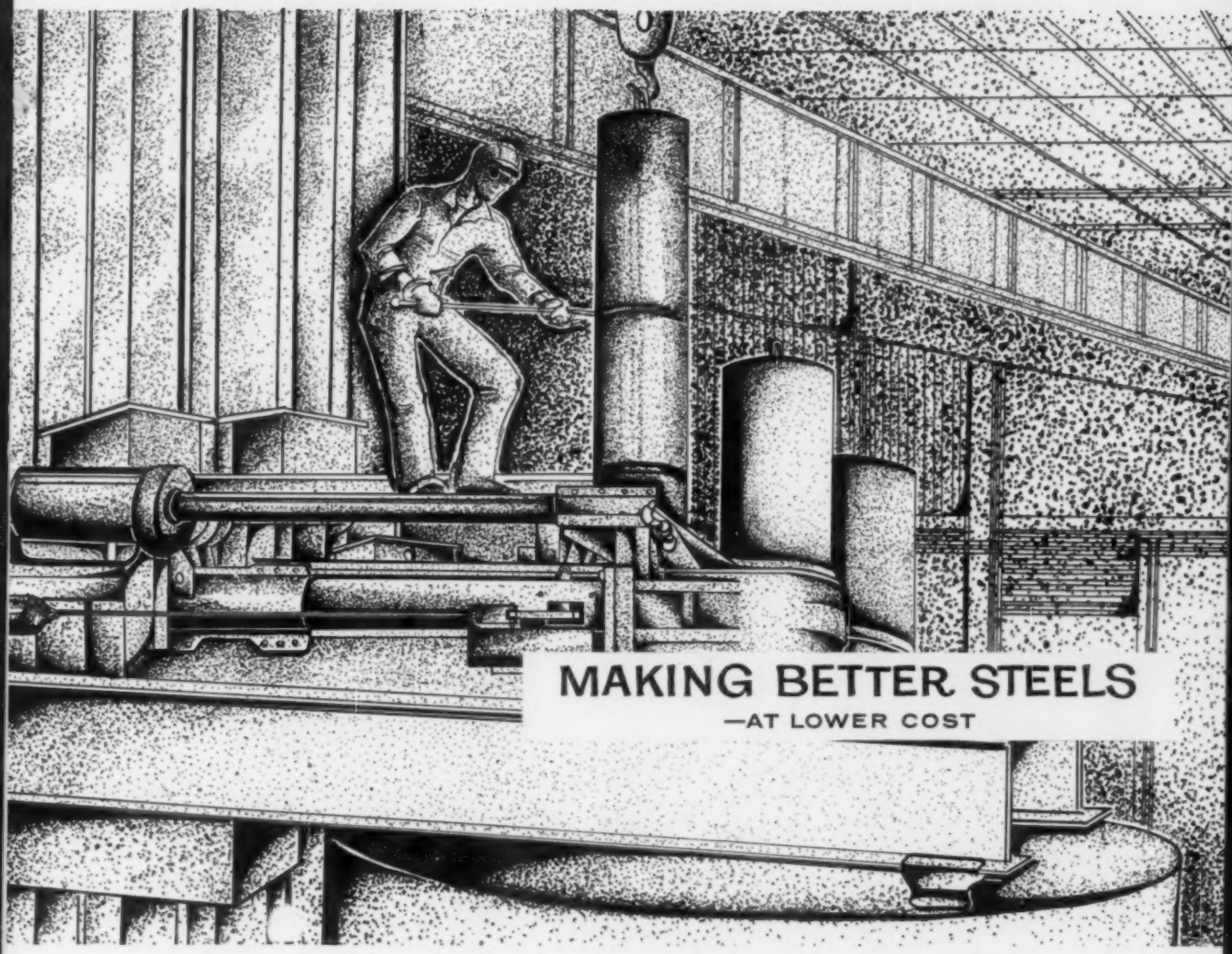

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Personnel

(Continued from page 312)

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(Continued on page 316)

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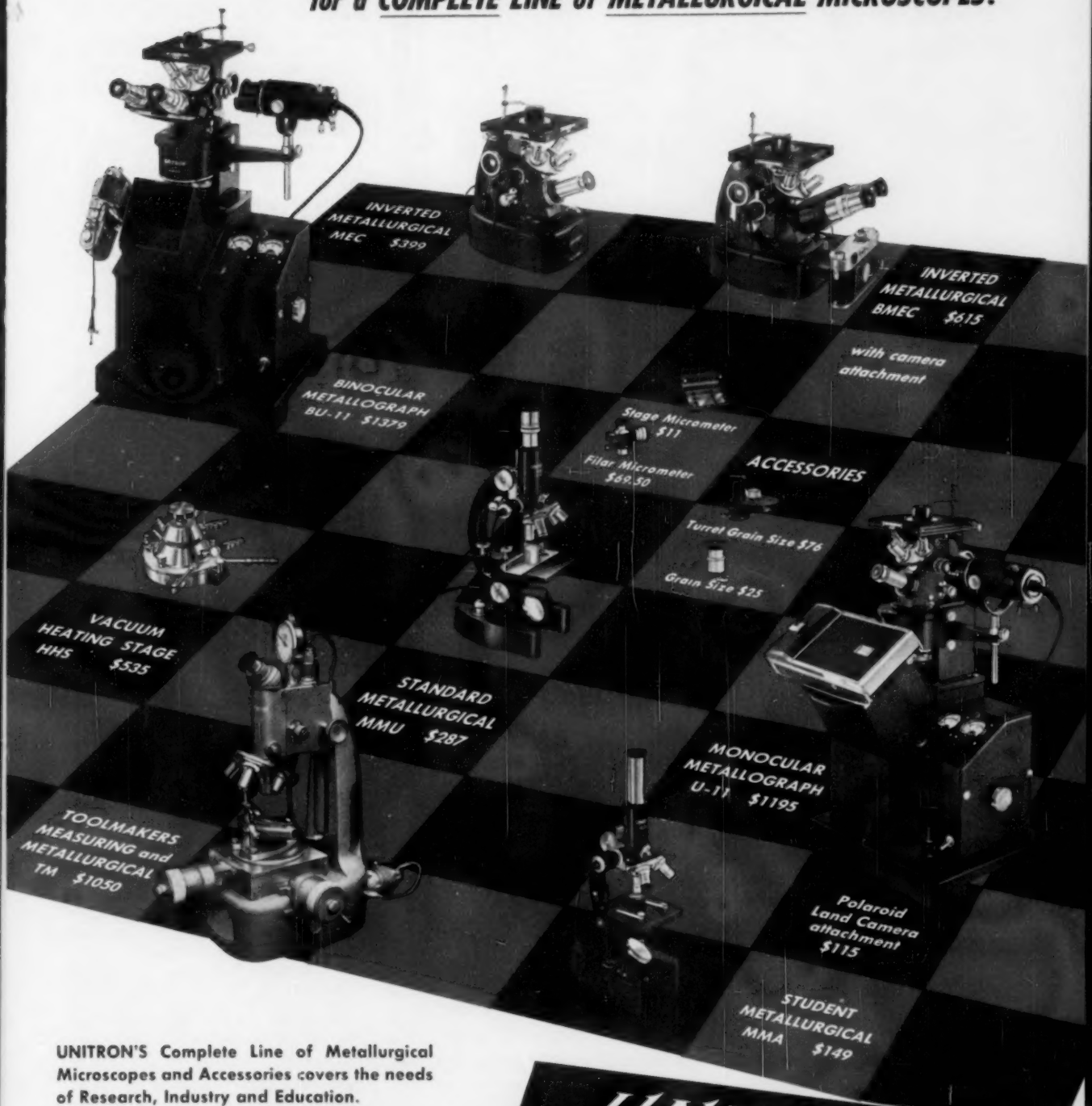

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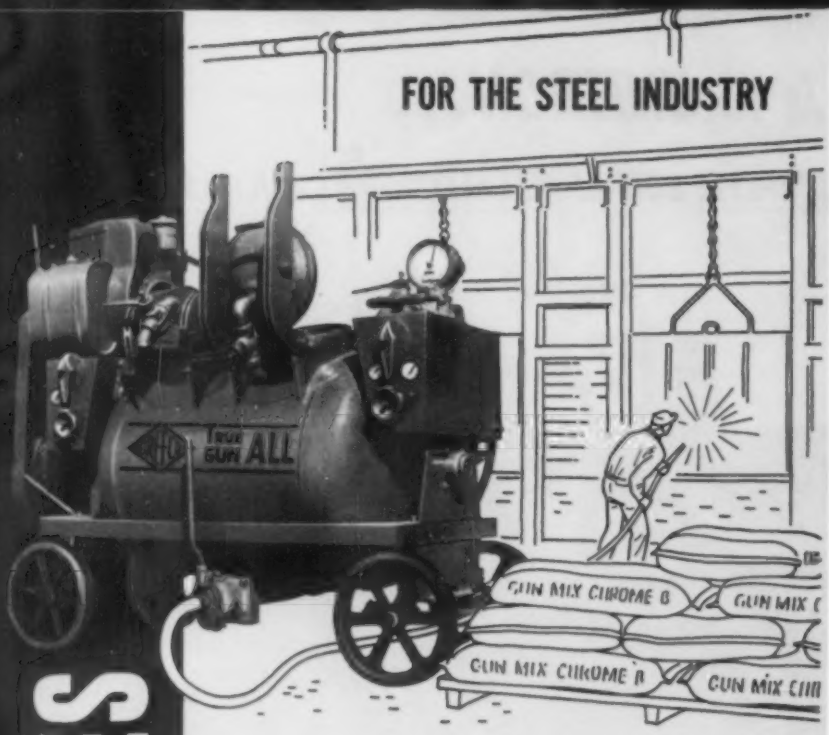

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Personnel

(Continued from page 314)

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The Iron and Steel Industry, No. 26, Vocational and Professional Monographs, by Tom Campbell, *Bellman Publishing Co.*, Cambridge 38, Mass., 40 pp., $1.00, 1957.—This Monograph deals with the career opportunities that are available in the iron and steel industry and shows the reader how to get into this vocation. Chapters in this self-help publication include: an over all view of the steel industry, which includes job descriptions; suggested educational preparation for jobs in steel; working in the steel industry—advantages and disadvantages; pay and working conditions in steel; and professional groups and trade associations.

Symposium on Steam Turbine Oils, ASTM Special Technical Publication No. 211, The *American Soc. for Testing Materials*, 1916 Race St., Philadelphia 3, Pa., 108 pp., $3.00.—Sponsored by ASTM Committee D-2 on Petroleum Products, this symposium was presented at the second Pacific Area National Meeting of ASTS, held in Los Angeles, September 1956. The eight papers in this conference cover the many aspects of the new demands imposed on turbine fluids. Some topics of the papers are: organizational and functional aspects of section on oil systems for turbines; rusting in turbine oil systems; antiwear requirements for navy turbine oils; monomolecular films of rust-preventative additives; and evaluation and performance of turbine oils. •

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Differential Thermal Analysis, by W. J. Smothers and Yao Chiang, *Chemical Publishing Co. Inc.*, 212 Fifth Ave., New York 10, N. Y., 530 pp., $16.00, 1957.—This illustrated work is the first survey of the methods of differential thermal analysis which will be of great interest to those engaged in metallurgy, chemistry, geology, soil studies and ceramics. The chapters include: origins of DTA; equipment; factors in qualitative DTA; quantitative DTA; and use and correlation of DTA results. Four appendices on references, numerical data, and other information of DTA complete the book.

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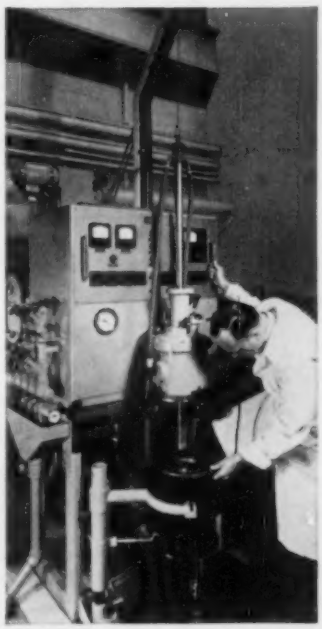

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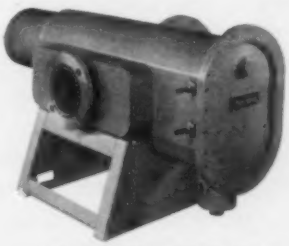

This Roots pump maintains low, vapor-free pressures. A motor operating within the vacuum drives its rotary frictionless pistons.

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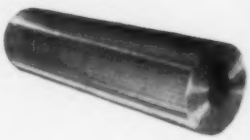

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levitation

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Typical levitation of alloy specimen in a ceramic-coated, specially shaped, radio-frequency induction coil.

melting of uranium-base fuel alloys

Electromagnetic levitation with simultaneous melting, pioneered by Westinghouse in 1949, is being investigated at Bettis as a tool for melting research quantities of pure uranium-base fuel alloys. Reactive metals such as U, Ti, V, Zr, Nb, Ta, and Mo cannot now be melted in any of the known refractory materials and still retain sufficient purity for precise metallurgical investigations. To avoid crucible contamination, Robert E. Droegkamp, senior engineer, has been developing a levitation melting technique for application to nuclear fuel development.

Levitation melting offers several advantages: each melting charge can be prepared easily by compacting powders or chips of the material; the specimen does not touch a crucible during heating or melting; the alloy is thoroughly mixed electromagnetically before chill casting; volatile impurities can be removed; and the molten alloy can be protected by a suitable atmosphere or vacuum.

The force necessary to levitate a given material is a function of the size, shape, and resistivity of the material, the size and shape of the induction coil, and the frequency and amplitude of the alternating current used. The high density and resistivity of uranium alloys pose problems. Instantaneous superheating causes evaporation and ionization that result in a short circuit to the coil. This allows the metal to cast itself.

To date, specimens of a uranium-base alloy weighing about 8 grams each have been levitated in the molten condition for periods up to 5 seconds at pressures up to 1×10^{-5} mm Hg. When this technique is perfected, it will be possible to prepare uranium alloys of higher purity than previously available for transformation, corrosion, phase diagram, and possibly irradiation studies.

This levitation melting process described briefly is only one of the many challenging projects which metallurgists encounter at Bettis. We welcome confidential inquiries from metallurgists interested in the excellent careers offered by the expanding nuclear power industry. U. S. Citizenship required.

Send your resume to M. J. Downey, Dept. #A-50, Bettis Atomic Power Division, Westinghouse, Box 1468, Pittsburgh 30, Pa.

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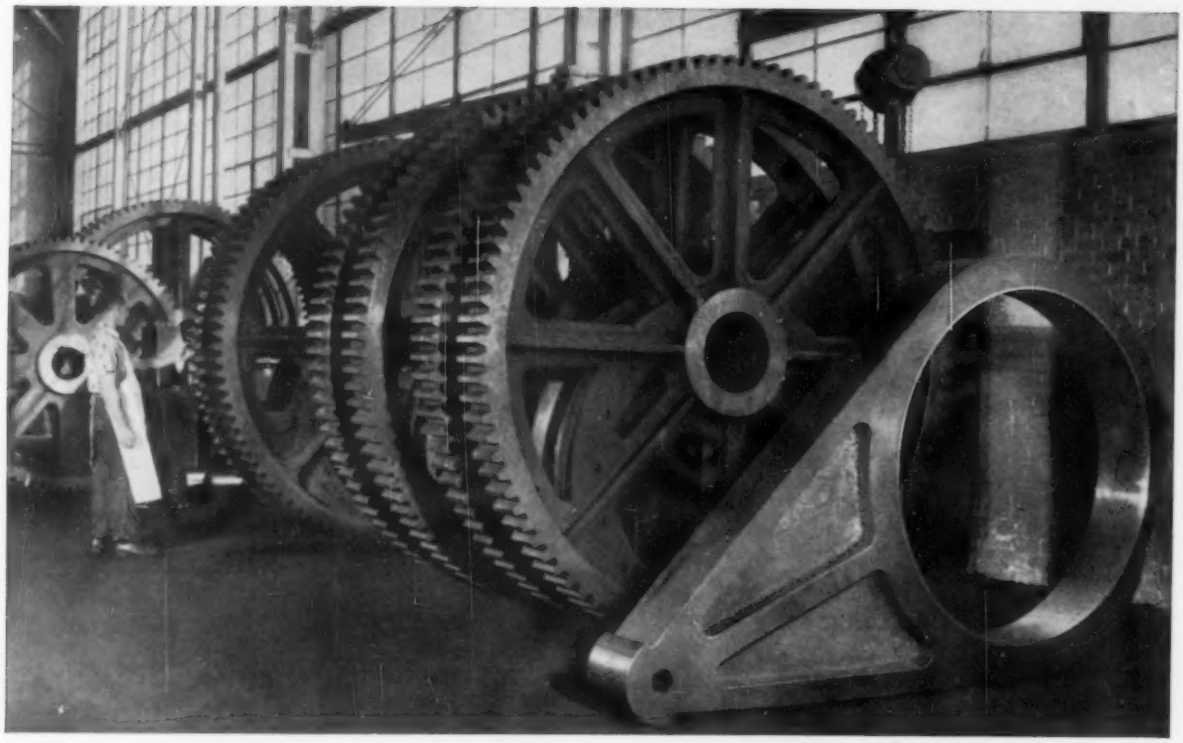

Dynamically stressed parts for 2000-ton "tryout" press...

Made of ductile cast iron, for superior strength, needed wear resistance

These large gears and connecting rod are used in a 2000-ton mechanical "tryout" press. In discussing his choice of ductile iron for these critical parts, Stewart Cumming, the designer – had this to say:

"We have for years favored cast iron for gear teeth and eccentrics because of its good wearing properties in bearing surfaces, but in some cases were forced to use either steel castings or weldments in order to obtain the necessary strength.

"The development of ductile iron enabled us to use this material to advantage, by providing the strength required for gear teeth without sacrificing good wearing surfaces on tooth faces.

"In the intermediate spur tooth gears and high speed herringbone gears of these large presses, good tooth-bearing surface qualities are particularly important. These gears are subjected to heavy stresses in clutching and braking.

"The use of ductile iron enabled the eccentric strap portion of the connecting rods to be held to as small a cross section as would be possible with steel – a distinct advantage in gaining operating clearances. The knuckle joint end, which is subject to high bearing loads at the time of impact, has better wearing properties in a bearing surface than could have been attained with an unheat-treated steel."

2000-ton "tryout" press (shown forming auto fenders) can handle most large dies or group of dies. Built by Federal Engineering Company, Detroit, a major builder of machinery, dies and tools, and designed by Stewart Cumming & Son, Ferndale, Michigan (engineers in the same line) with ductile iron in key operating parts.

More ductile iron advantages...

Other designers report additional ductile iron advantages for gears: excellent machinability, high damping capacity, and good castability. Surface hardening techniques can usually be used, too, to increase hardness to 600 BHN.

You'll find all these subjects and more covered *in detail* in Inco's new "Ductile Iron Digest." Send for one.

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ductile iron...the cast iron that can be twisted and bent.

Columbium, Important Nuclear Metal

Recently, the AEC released a review by W. D. Klorp and C. R. Tipton of the Battelle Memorial Institute, describing significant developments in materials used in reactors. Columbium is particularly important because of its moderately low cross-section for absorbing neutrons (six times that of zirconium, but less than a half that of most other high-temperature materials), its high temperature strength (which exceeds zirconium), its ability to form useful alloys with uranium, its resistance to corrosion by some liquid metals and some gases, and the ease with which it can be fabricated. Drawbacks are its high cost and poor corrosion resistance to many materials. However, greatly increased production should lead to lower costs, but probably not equal to zirconium, because of problems involved in the processing of low-grade columbium ores. For "A Columbium Primer," see page 340.

High-Purity Columbium

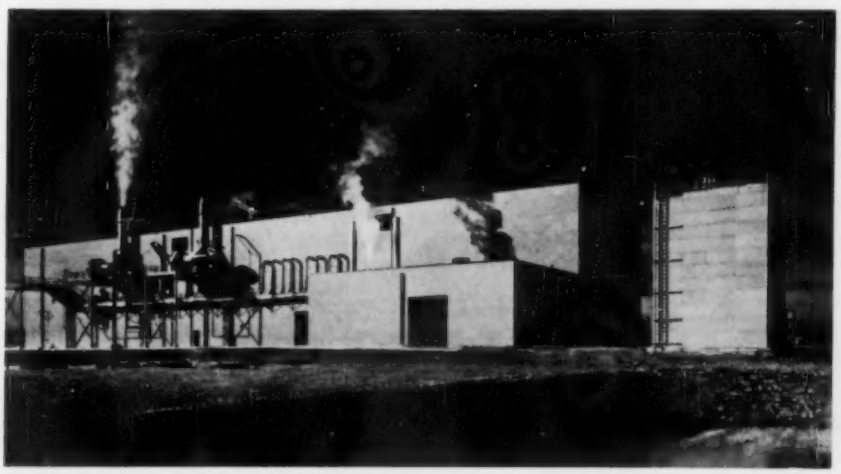
Refinements in production techniques have enabled Electro Metallurgical Co., Div. of Union Carbide Corp., to increase the purity of its columbium metal to 99.6 pct, which is reported to be the highest purity of columbium commercially available. The new specifications are as follows: 99.6 pct Cb, 0.10 pct O_2, O.O4 pct N_2, 0.05 pct C, 0.15 pct Ta, 0.01 pct Ti, 0.01 pct Fe, 0.001 pct Si, and 0.0001 pct B. The metal is produced in the form of roundels, small cylindrical shapes, selling at up to $70 per lb.

New Columbium-Tantalum Pilot Plant

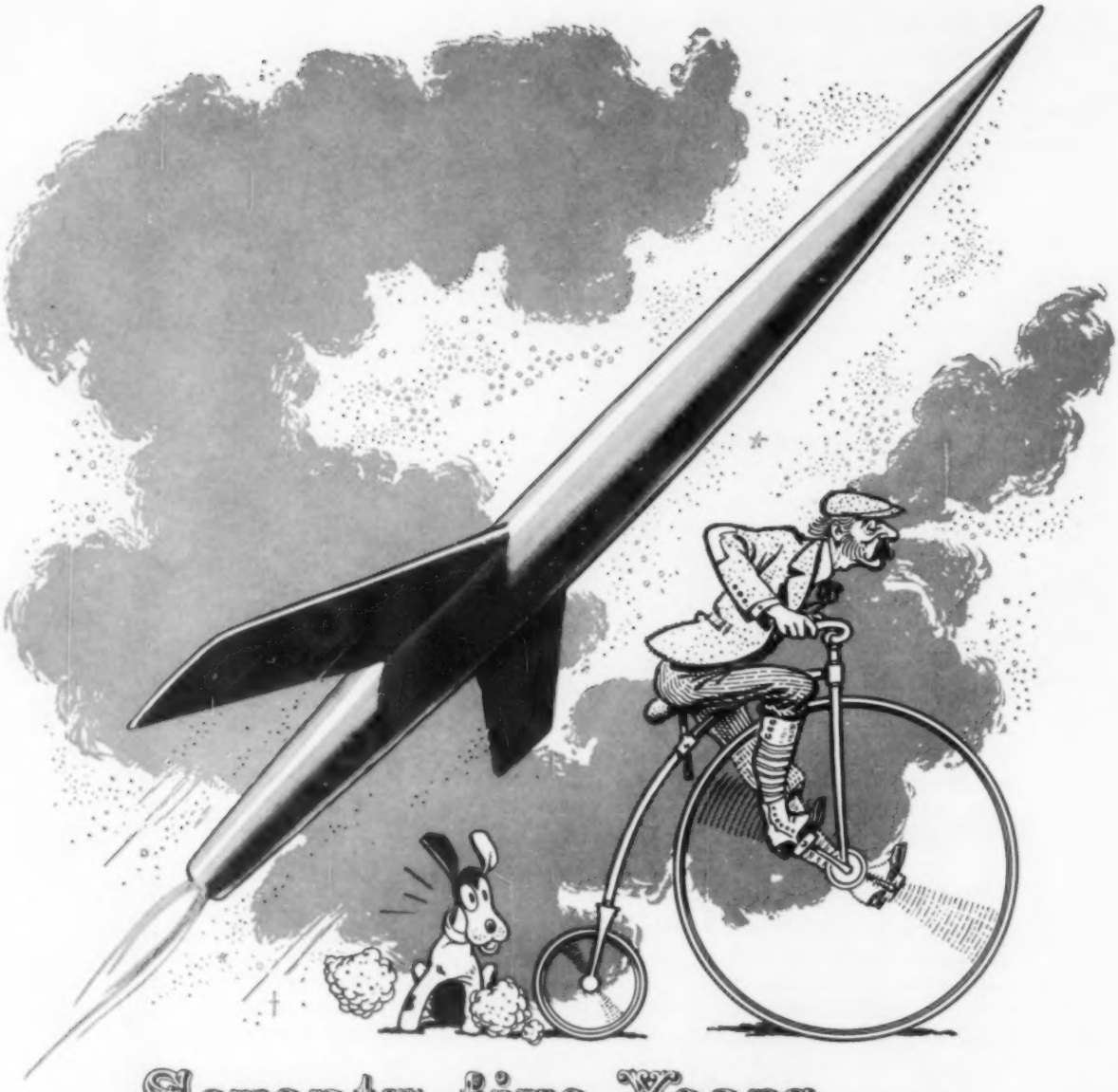
At Cincinnati, Ohio, U. S. Industrial Chemicals Co. has begun construction of a pilot plant to produce tantalum and columbium at the rate of 3 tons per month when operating on a three-shift basis. The sodium reduction process—similar to that now in use at Ashtabula, Ohio, zirconium plant of Mallory-Sharon Metals—will be used in the pilot plant.

Columbium-Tantalum Commercial Plant

Operations are underway at Fansteel Metallurgical Corp.'s new $6.5 million tantalum-columbium plant at Muskogee, Okla. Primary reason for the plant's construction has been the increased demand for tantalum capacitors of which Fansteel produces a variety. The plant treats tantalum-columbium ores of wide geographical origin to produce metal powder which is compacted into bars and vacuum melted.



Muskogee, Okla., plant of Fansteel Metallurgical Corp. Shown is the Chemical Operations building with equipment for converting ores into tantalum and columbium metal powders.



Seventy-five Years ◇◇◇

TEN MILES an hour was "speed" in 1883 when Wyman-Gordon started to make forgings for the high-wheel bicycle. Through the 75 intervening years forgings have made important contributions to the phenomenal advances in propulsion. Progress from the first "horseless carriages"...from the early "flying machines"...to the supersonic speeds of today ...would not have been possible without forgings produced by the most advanced techniques of the day.

Wyman-Gordon is proud of its achievements in these fields and, as the largest producer of automotive and aircraft forgings, is prepared to accept the challenge of the future. Today, as for 75 years, there is no substitute for Wyman-Gordon quality and experience.

WYMAN-GORDON COMPANY

Established 1883

FORGINGS OF ALUMINUM • MAGNESIUM • STEEL • TITANIUM

WORCESTER 1, MASSACHUSETTS
HARVEY, ILLINOIS • DETROIT, MICHIGAN

Columbium vs Niobium:
The Readers Take Over

IN the October 1957 issue of JOURNAL OF METALS, the editorial page treated the controversy which exists over the use of columbium or niobium. At that time, columbium was established as the name that would be used in this magazine, following historical fact and the industry which uses the metal commercially, but in spite of decision to the contrary by the International Union of Pure and applied Chemistry and the American Chemical Society. (ACS).

The response to this editorial has been quite startling; most, but not all, has been on the side of columbium. Now it is not the desire of the AIME or the JOURNAL OF METALS to stir up dissent within our sister societies, but we were curious as to how the members of the ACS felt about their society's stand on the subject, and we queried Mr. H. G. Tanner, an ACS member, on the subject. Mr. Tanner's comments shed a considerable amount of new light on the subject, and are printed below as received:

"Fifty three years ago Edgar Fahs Smith[1] (three times president of the American Chemical Society) stated: 'Marignac, after a careful study of a number of minerals from various localities, announced the the existence in them of but two elements—the columbium of Hatchett and the tantalum of Ekeberg. —Marignac's conclusion was accepted by the chemists of the world as final.' Smith recommended that the name columbium be adopted officially by the ACS, and his recommendation was duly approved.

"In 1951 the Council of the ACS decreed that niobium should henceforth be the name of element 41 because the IUPAC at a meeting in Amsterdam, 1949, voted in favor of niobium. When the ACS announced this, we, the dues paying members-at-large were astounded. The Sections had not been asked beforehand to express their views. No reason was issued with the decree other than that the IUPAC preferred niobium.

"No published statement has been found stating why IUPAC preferred niobium. If the preference were decided by popular vote, the trivial representation from the United States would never have been noticed.

"E. J. Crane,[2] former chairman of the ACS Committee on Chemical Nomenclature, stated that ACS went along with IUPAC 'in the interest of international unification.' Presumably he meant unification of chemical nomenclature, and this is a desirable goal. Unification, however, need not always be arbitrary. Science has provided a principle that has long been applied, whenever possible to do so, for achieving uniformity of nomenclature. That principle is: He who makes a discovery shall be entitled to name it. This principle was applied by Edgar Fahs Smith, J. W. Mellor, a former Council of the ACS, and others for choosing between columbium and niobium. Apparently the 1951 ACS council was more interested in achieving uniformity with IUPAC than unification of nomenclature by the normal procedures of science.

"The name columbium has been in use for more than 150 years, and in the United States columbium has been used exclusively from the early 1900's to 1951 when the ACS changed its mind. During this half century more research and development of columbium occurred in the United States than in all other nations combined. In 1955 fifteen nations shipped nearly 10,000,000 lb of columbium ore to us. Columbium has become part of the language of industrialists, miners, stevedores, bankers, prospectors, clerks, welders. Consequently, the name columbium is here to stay. The name columbium is not simply a matter of chemical nomenclature. Therefore, it cannot be eliminated by those who think only in terms of chemical nomenclature, and it must not be changed if a fundamental principle of justice is to be retained."

[1] Edgar Fahs Smith: Proc. Amer. Phil. Soc., vol. 44, p. 151, 1905.
[2] E. J. Crane: Chem. Eng. News, vol. 31, p. 1577, 1953.

H. G. Tanner
Frederick, Md.

Unsolicited comments on the editorial have been largely pro-columbium, and a sampling of these is reprinted below:

. . . .I personally am in complete accord with retaining the names columbium and tungsten and have not given much thought to the nonsense of changing these names. . . . Will the AIME editors continue to use the name columbium? Do the editors have any information on the policy of . . . other technical magazines?

M. J. Day, V. Pres.
Research and Development
Crucible Steel Co. of America

The editors will continue to use columbium as the proper name. If the AIME should adopt an official view to the contrary, we would of course yield. As to the view of other magazines, see below:

. . . .As you know, *Materials in Design Engineering* is strongly in favor of retaining the name, "columbium." As a member of AIME, I am also in favor of continuing to use columbium as the designation. . . .Just a few weeks ago I received a call from Cleveland from a man who wanted to know something about the new element, niobium. When I told him it was just another name for columbium, he was quite unhappy over having made the call, since he is familiar with . . . columbium. I certainly hope the AIME will join the Welding Society and the ASM in using the original name "columbium" for the element.

John L. Everhart
Technical Editor
Materials in Design Engineering

. . . .I cannot help but believe that acceptance in America of the efforts of the International Union of Pure and Applied Chemistry to standardise the name of Element 41 will be in the best interests of the majority of people and in the best interests of all in the long run.

E. J. Crane
Director and Editor
Chemical Abstracts

Mr. Crane expressed the case for niobium in an article in Chemical and Engineering News *on page 44 of the January 13, 1958, issue.*

. . . .I am writing to say that I heartily agree with your suggestion that the name columbium be retained.

Russell Franks, Mgr.
Marketing Research Div.
Electro Metallurgical Co.

. . . .As you point out, the metals industry. . . .is strongly in favor of using the term columbium; and if AIME and ASM will stay with the term columbium, I am certain that in a relatively short time the U. S. A. will soon be rid of the intruder term niobium except when foreign literature will be encountered. Congratulations. . . We are with you.

A. de Brasunas, Director
Metals Engineering Institute

In the spirit of fair play, it is incumbent upon us to also print the following:

. . .You can imagine my surprise . . .when I read the article on the Reactive Metals Conference on page 143 of the February issue. Here I thought I had . . . staunch supporters of columbium versus niobium and lo and behold, the list of metals to be covered included niobium. It looks as though subversives are at work in your camp.

L. F. Granger, Mgr. of
Advertising
Electro Metallurgical Co.

Our apologies, Mr. Granger. We shot our copy editor and hope that it won't happen again.

—The Editors.

furthering creative technology at

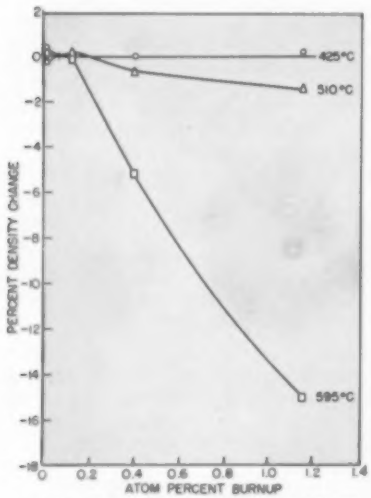
The KNOLLS ATOMIC POWER LABORATORY

The significant relationship between advances in materials and processes, and advances in nuclear propulsion systems has generated a continuous program of original investigations in metallurgy and ceramics at The Knolls Atomic Power Laboratory.

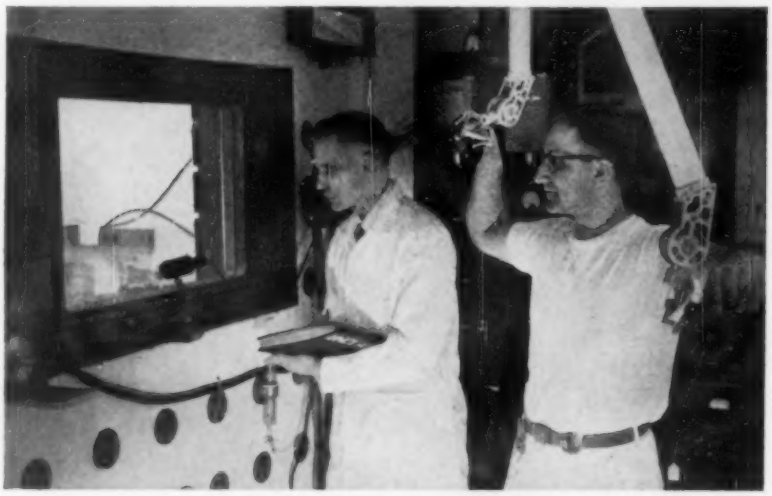
Currently the Laboratory is working toward the design of power reactors for operation at higher temperatures and with higher fuel burnups. A typical materials problem is the swelling and blistering of fuel elements caused by the agglomeration of inert gases (Kr and Xe) formed during nuclear fission.

• • •

An example of the physical-metallurgical studies now under way in this area is the experimental work described below.



DENSITY CHANGES IN ZIRCALOY CLAD 8 w/o URANIUM-ZIRCONIUM ALLOYS ON POSTIRRADIATION ANNEALING



Dr. W. V. Johnston (left) recording data at post irradiation annealing cell.

POST-IRRADIATION ANNEALING OF URANIUM-ZIRCONIUM ALLOYS

To obtain information on the agglomeration mechanism and the extent of damage experienced, specimens of an 8 wt % enriched uranium-zirconium alloy which had been irradiated to a maximum of 42% of the U-235 atoms fissioned were annealed in vacuum furnaces at 425, 510 and 595°C. Percent density changes were determined as a function of temperature at an annealing time of 500 hrs. As can be seen in the graph, specimens of all burnups were stable at 425°C but volume increases of up to 15% were obtained at 595°C.

Additional information on the amount and rate of Kr and Xe diffusion and release from the fuel elements has been obtained by using β and γ counting techniques. The effects of irradiation and annealing on the temperature of phase transformations and on alloy structures are being studied by measurement of electrical resistivity, and use of advanced metallographic techniques.

PROFESSIONAL OPPORTUNITIES

There are openings at the Laboratory for metallurgists and ceramists interested in development, application and fabrication of nuclear core materials. Degree is required; advanced degree and/or related experience preferred. (U.S. Citizenship) If you qualify, please send resume, including salary requirement, to: Mr. A. J. Scipione, Dept. 37-MQ.

Knolls Atomic Power Laboratory

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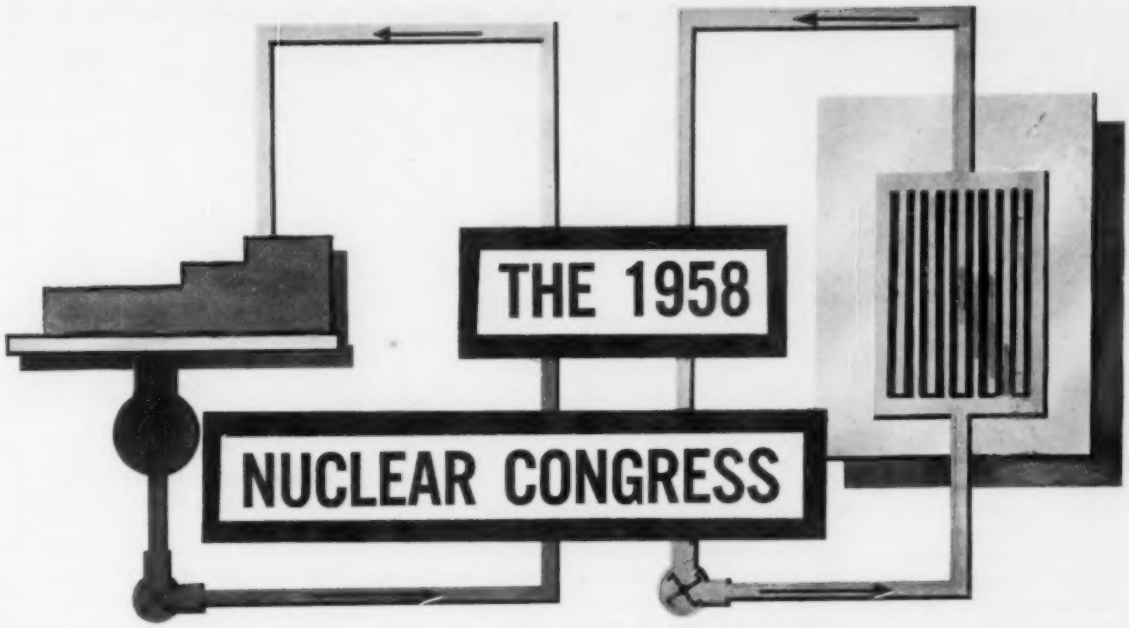


A summary of the highlights of this year's meeting, which included the Nuclear Engineering and Science Congress, the Hot Laboratories and Equipment Conference, the Atomic Energy Management Conference, and the Atomfair.

by J. J. Burke

BOTH optimism and pessimism were very much in evidence as the 1958 Nuclear Congress convened in Chicago in March. General business conditions were responsible for a reduction in size of many of the exhibits, and also cut into NESC registration, which was 1932, down 4594 from last year.

During the year since the last Congress, many companies have gotten *cold feet* in the nuclear power field. *Economic* nuclear power seemed too problematical to warrant capital expenditures at this time.

However, the best informed views at this year's Congress reflected a cautious optimism. John R. Dunning, Dean of Columbia University's School of Engineering and a man associated with atomic energy since the days of the Manhattan Project, feels that prospects for economic nuclear power are much better this year. "It's true", he said in an interview with this reporter, "that we might have overstated the case in order to get companies interested in atomic power at all. Costs over five times as much as conventional power stations at Shippingport scared a lot of people, but that was on a cost plus basis, and it is not indicative of what can be done. Results in the last year with boiling water reactors at Vallecitos and Argonne indicate a much more favorable picture." Dr. Dunning concluded by saying that, although economic atomic power will not be here this year or next, it should "definitely" be a reality within five to ten years.

VBWR called highly successful

Operation of the Vallecitos Boiling Water Reactor (VBWR) was described by J. E. Maider, of G. E.'s Atomic Power Equipment Dept. in San Jose, Calif., at the Atomic Energy Management Conference.

Mr. Maider's remarks were predicated on the fact that the Vallecitos reactor was not set up purely as a power producer. It is equipped for either single or dual cycle operation, and its main function is to test out components that will be utilized in the full-scale 180,000 kw Dresden power station to be built by G. E. for the Commonwealth Edison Co. of Illinois and the Nuclear Power Group.

Mr. Maider's comments reflected considerable pride in the speed with which the reactor was completed; the unit went on line at full power less than 17 months after ground-breaking.

Developmental work being performed on the reactor falls into six categories: fuel data, stability, operation under utility-type loads, operation with radioactive steam throughout the system, refueling techniques, and power output data.

Stability of the VBWR, along with ability to handle utility-type loads, have been the most outstanding characteristics of its operation. The reactor has been operated at pressures up to 1000 psi at an output of 30,000 thermal kw with no sign of instability, and an almost instantaneous change of 25 pct of the full load resulted in a pressure variation of only 10 lb.

Through the use of high-purity water, and a turbine design that eliminates any *pockets* and insures high velocity and washing, it was found that direct contact maintenance was completely practical after 900 hr of operation.

As with many experimental reactors, it has been found that the VBWR is capable of exceeding its licensed output. The original 20,000 thermal kw has been boosted to 30,000, and it is felt that further rises are entirely possible, and 100,000 thermal kw could be achieved with only minor modification.

Experience at Argonne leads to $180 per kw cost figure

Results at Argonne National Laboratory's Experimental Boiling Water Reactor (EBWR) have led to a prediction of $180 per kw as the ultimate installed cost of a boiling water power system. This prediction was voiced by B. I. Spinrad, Senior Physicist at Argonne. Dr. Spinrad based his prediction on experience with the EBWR in several categories: construction experience and power ratings, fuel cycle experience, normal operation, and plant hazard. His discussion of these areas made the following points:

"The first of these items is concerned with the question of predicting an installed cost per thermal kw. Our experience has indicated that the EBWR, considering only its nuclear portion, is capable of 100 thermal megawatts output at an installed cost of about $4,500,000. The 100 thermal megawatt capability is admittedly an extrapolation; the cost is firm, and would be on the high side except for realization of the fact that an experienced development laboratory can often cut costs through use of skilled labor of a type not generally available at the pro-rated salary scale. In opposition to other reactor types, the size factor is not as important in light water boiling reactors. Hence, in extrapolating to, say 600 thermal megawatt sizes, the usual 0.6 power rule can be taken more as an experience factor. Using this rule, I would get $22 per kw of heat as installed thermal cost. To this must be added superheating costs, which might come as high as $100 per kw of heat at 15 pct added heat. This finally gives $31.3 per thermal kw at 28 to 29 pct generating efficiency; or an installed cost of about $115 per electrical kw for the heater only. Adding $65 per kw for generating equipment, one gets a predictable $180 per kw as the ultimate installed cost for boiling reactor power systems. This is a little higher than our modern fossil plants."

Economic power awaiting lower cost fuels

A lower fuel cost is the goal that scientists are shooting at to bring that $180 figure down. There are three avenues for pursuing this goal: an improved lattice with higher enrichment, reduction of fabricating costs, and recycling of the plutonium or U^{233} formed. All three methods are being actively studied.

The reprocessing problem was discussed by J.W. Ullman of the Oak Ridge National Laboratory, and, in his opinion, the all-aqueous method will probably receive more attention than other methods for at least the next several years. However, Dr. Ullman stated that cost reduction would be predicated on several big "ifs," including standardization of fuel elements, expansion of the nuclear power industry, development of more corrosion resistant process equipment, and a market for fission and transmutation products.

Nuclear Engineering education

A special session was devoted to educational problems at this year's Nuclear Engineering and Science Congress, with several papers contributed by the American Society for Engineering Education.

The general feeling at this session was that nuclear engineering is just one more parameter to be dealt with, to be fitted into the other basic engineering sciences. Specialties in nuclear engineering, it was felt, should be left for graduate studies.

Glen Murphy, of Iowa State College, discussed the problems posed by such integration, and offered two possible solutions:

"For the most part, the colleges have taken the stand that it is far better to teach the student the fundamentals and basic principles than it is to indoctrinate him with detailed technology that will be obsolete by the time he graduates. While this is a general problem, it is particularly acute in the field of nuclear engineering for two reasons. One is that the technology is developing so rapidly that those design details the professor hears about at a meeting or sees at a plant are obsolete by the time he can return to the campus to tell his students. The second point peculiar to nuclear engineering is that the graduate must be well grounded in conventional engineering fundamentals because he is first of all an engineer. In addition to the conventional fundamentals he must acquire the fundamentals of nuclear transformations, radiation and its interaction with matter, and reactor science. The net result is that design details, such as values of thermal conductivity for liquid metals or standard sizes of aluminum tubing, are not going to be included. Such items are best learned on the job anyway."

Who should do detail work?

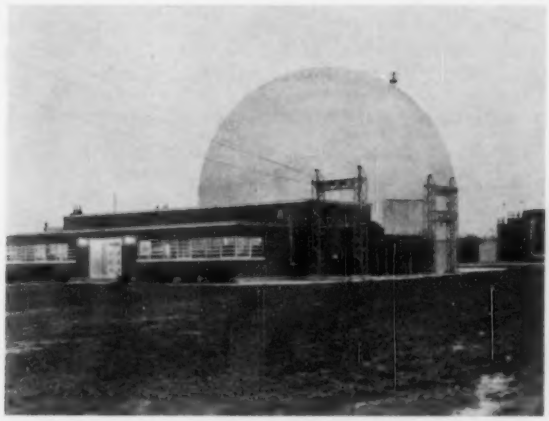
"There are two approaches to overcoming the shortcomings of the four, five, or six year graduate as a detail designer. One is to give him the indoctrination in detail design on the job and along the lines approved by the specific company (which may not be approved by any other company). The indoctrination may take place when the man first reports for work and may require several months of training or close supervision. However, at the end of that period the man will be more valuable and have more potential than he would if that much time had been taken from his fundamentals, while in college, to give him detail design.

"The second approach is not to use the four-year graduate engineer for detail design, but to use him for conceptual design, system analysis, and similar activities for which his broader training fits him. It has been well demonstrated that the graduate of a qualified technical institute or a special two-year college program can do detail design as effectively and probably more happily than the graduate of a four-year program. . ."

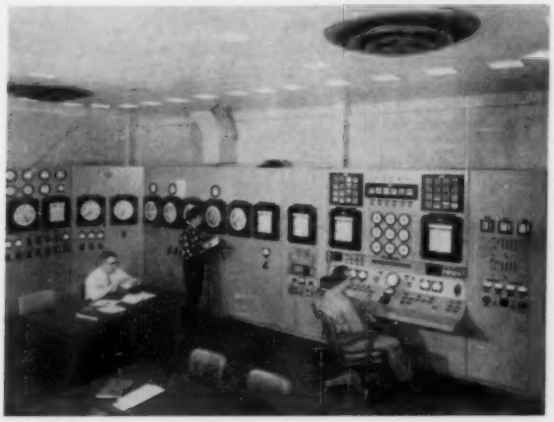
Dr. W. Kenneth Davis, of the AEC, gave emphasis to the variety of technical skills needed in nuclear projects with the following figures: ". . .of the 7000 to 7500 scientists and engineers employed on such [atomic energy] programs in 1956, the discipline of mechanical engineering led all the rest. The so-called nuclear engineer was only a small portion of the total, . . . 500 in all, even by their own designation. There were large numbers of electrical and electronic engineers, chemists and biochemists, metallurgical engineers, civil engineers, instrument engineers, mathematicians, and statisticians and so on. All of these professions make up the engineering and scientific force of the nuclear programs."

Dr. Davis concluded his remarks by calling on industry to help establish research and education programs in this new field, for it is only through such cooperation with the AEC and the schools that a structure can be built that will "integrate nuclear science and engineering into our conventional curricula."

The nuclear industry, it might be said, is coming of age. An increasing body of knowledge is pin-



The Experimental Boiling-Water Reactor at Argonne National Labs, Lemont, Ill. Originally licensed for 20,000 thermal Kw, it was later doubled and is now rated at 62,000 thermal Kw.



Control room of the EBWR. At left are controls for the steam power generating equipment; the nine dials above the console show the position of the control rods in the reactor.

pointing the problems that need further work, and as more is learned, the hazards are lessened. This can only lead to a more successful exploitation of this new form of energy.

Nuclear radiation and Radioisotopes in metallurgy

by M. T. Simnad

Application of nuclear reactions to metallurgical research problems is well known, but it is conceivable that some researchers might not be aware of applications in a particular field. Dr. M. T. Simnad, formerly with the Carnegie Institute of Technology and now Chairman of the Metallurgy Dept. at General Atomics, a Division of General Dynamics Corp., is a man well versed in these applications, and he presented a discussion of the subject at this year's Atomic Energy Management Conference. His talk is summarized below:

The application of nuclear irradiation and radiotracers in metallurgy has been increasing in recent years with the availability of irradiation facilities and the mass production of radioisotopes. A recent review of the subject by the author included reference to about 500 papers. Nuclear irradiation has been employed both as a means to detect minute quantities of impurities or minor constituents and as a unique tool to study the structure of metals. Radioisotopes have been used in almost all branches of metallurgical research.

Nuclear Irradiation

1) Activation Analysis: Impurities and minor elements in metals and alloys may be identified and their quantities determined, if they yield radionuclides having suitable half-lives and measurable radiations. The sensitivity of detection is governed primarily by the activation cross-section, the flux and energy of the bombarding particle, the time of irradiation, and the half-lives of the radionuclides obtained.

2) Irradiation Effects: The study of irradiation effects has helped to elucidate problems related to the structure of metals, especially the behavior of vacancies and interstitials. The properties which are affected by radiation include: electrical and thermal conductivity, phase transformations, mechanical properties, and corrosion resistance.

Radioisotope Applications

1) Segregation: The inhomogeneity in alloys has been investigated by means of autoradiography of active specimens. The distribution of any constituent may be determined on the autoradiograph, either by adding a radioisotope of the element when preparing the alloy, or by irradiating the prepared specimen.

2) Friction and Wear: One of the sliding metal surfaces is here made radioactive prior to the friction experiment, or the transferred metal is activated *in situ* after the test. Autoradiography of the surface will give a quantitative measure of the distribution of the transferred material.

3) Vapor Deposition and Vapor Pressure: The mechanism of heterogeneous nucleation of metals from the vapor phase has been studied with the aid of radioisotopes, which have provided a convenient means for studying the condensation process.

The thermodynamic properties of alloy systems can be calculated from vapor-pressure measurements, and the range of such measurements has been greatly extended by use of radiotracers.

4) Slag-Metal Reactions: The use of radiotracer techniques has proved to be of value in studies of the physical chemistry of slag-metal reactions, such as the dephosphorization and desulfurization of steel.

5) Corrosion and Surface Reactions: Various types of electrolytic action on metal surfaces in aqueous solutions have been studied by means of tracer methods, including corrosion and passivity, electrochemical exchange, and electrodeposition.

The mechanism of high-temperature oxidation of metals and alloys may be determined most directly with the aid of radioisotopes. The tracer technique is employed to determine the diffusion rates in the oxide films and to identify the diffusing species by means of marker movement experiments.

6) Diffusion. The results of tracer measurements of diffusion in alloys have been more reliable than those of chemical diffusion. The unique use of tracers to measure self-diffusion rates in metals has contributed immensely to the understanding of diffusion processes.

NEW METALS FACE THE FUTURE

Industries now being born demand metals capable of operating at higher temperatures, of higher corrosion resistance, and of particular nuclear or electrical properties. Here is presented a provocative survey of where we stand now, and what the future may hold in store, as research and development proceed.

by C. M. Brown and R. W. Fountain

IN recent years, the automotive, aircraft, atomic energy, electrical, oil, chemical, and electronic fields have emerged as new basic industries. Industrial developments associated with these industries resulted in demands for metals capable of operating at higher temperatures and stresses, materials with greater corrosion resistance, materials that are either transparent or opaque to neutrons, and materials with new or improved electrical properties over an extremely broad temperature range. In metals that were considered to hold promise, there was the need for higher degrees of purity comparable to levels usually available only in the laboratory. New metals that have achieved stature are titanium, columbium, molybdenum, chromium, zirconium, tantalum, vanadium, and silicon.

Materials for high-temperature use

The major impetus to the demand for materials of enhanced resistance to creep at elevated temperatures is due to development of gas turbine engines. But in the steam power-generating industry, operating temperatures have increased from about 800° to above 1200°F, while operating stress has increased from about 1300 psi to about 5000 psi. In the petroleum and the chemical processing industries an increase in temperature and pressure is required for production of gasoline from petroleum, hydrogenation of coal and oil, ammonia synthesis, and production of polyethylene, to mention only a few. In the field of atomic energy, the need for materials to withstand high temperatures and stress and corrosive conditions is an increasingly pressing demand.

Further improvement in efficiency of many industrial processes depends largely on the continued improvement in creep-resistant alloys. In the case of the gas turbine, for example, efficiency can be increased in two ways. Compression could be increased at existing gas temperatures, which would require materials of higher creep resistance at existing engine temperatures, or the gas temperature could be increased, which would necessitate new materials with the same resistance to creep at higher temperatures as existing alloys exhibit at current operating temperatures.

Optimum quality requirements

Materials for high-temperature applications must meet a variety of requirements involving their mechanical, chemical, and physical properties. They must resist static and dynamic stress, i.e., exhibit adequate room and high-temperature strength, creep resistance, fatigue strength, toughness, and a good relationship of these properties to density. The allowable total deformation during service life is restricted, although an appreciable ability to deform is often desired to prevent the possibilty of sudden and undetected initiation of fracture. Corrosion and oxidation resistance must be adequate. To prevent deterioration of properties and initiation of fracture, the metallurgical structure of the metal or alloy must be sufficiently stable during service life. Finally, hot-working or casting, heat-treatment, machining, and welding must be fairly easy and the price economically feasible.

The optimum of all these requirements cannot be provided simultaneously by a single alloy. The order

C. M. BROWN and R. W. FOUNTAIN are with the Fine Metals and Chemicals Dept., and the Metals Research Laboratories, respectively, of Electro Metallurgical Co., Div. of Union Carbide Corp., Niagara Falls, New York.

of importance of the property requirements and the most desirable combination of properties is determined, therefore, by the actual service application.

Maximum service temperature's relation . .

The maximum service temperature for various classes of high-temperature alloys is based arbitrarily on a life of 100 hr at 20,000 psi in a conventional stress-rupture test. Aluminum-base alloys find a maximum service temperature under the conditions specified of about 500°F, while titanium can be used at the present time to roughly 900°-1000°F, and the iron-base (both austenitic and ferritic) materials leave the picture around 1350°F, but all enjoy good oxidation resistance, thermal shock resistance, and high ductility. Cobalt and nickel-base alloys extend service temperature to about 1600°F.

. . . to melting point

The results of recent research suggest that the maximum service temperature (T_s) at which a material exhibits an adequate engineering strength (i.e., 100 hr life at 20,000 psi) may be related to its melting point (T_m).[1] For pure metals, the ratio between the so-defined maximum service temperature and the melting point ($T_s:T_m$) is about 0.4, when both temperatures are expressed in degrees Kelvin (0°K = −459.4°F). This ratio is effectively increased by alloying, the upper limit being approximately 0.8. In a study of this relation for pure nickel and three nickel-base high-temperature alloys, it was found that the service temperature of pure nickel, 780°F or ($T_s:T_m = 0.40$) is increased to about 1740°F or ($T_s:T_m = 0.75$) in a nickel-chromium-tungsten-titanium-aluminum alloy. This latter temperature appears to be close to the upper limit for nickel- or cobalt-base alloys.

This empirical relationship finds additional support from consideration of the recrystallization temperatures and the diffusivity data for pure metals and alloys.[2] It is well known that a high recrystallization temperature is a prerequisite for good creep-resisting properties. Data have been reported that indicate that for pure metals, the recrystallization temperature is about 0.4 of the melting point, when both temperatures are on the absolute scale, Fig. 1. These data suggest that the upper service temperature for pure metals may coincide with their recrystallization temperature. The unique position of columbium, molybdenum, tantalum, and tungsten having recrystallization temperatures well in excess of 1000°C is clearly evident.

. . . used to extrapolate upper service temperature

If this relation is valid, what then might be expected as an upper service temperature for the other common alloy bases discussed? To be rather realistic, assume a ratio $T_s:T_m = 0.6$, which should take into account the fact that the addition of alloying elements to base metals will most probably result in a decrease in their melting point. It should be remembered that this idea was developed using nickel and nickel-base alloys which have a face-centered cubic crystal structure which usually possesses better high-temperature resistance to deformation than either body-centered cubic or close-packed hexagonal. Also, problems of oxidation have not been taken into account. The new picture for maximum service temperature, based on the $T_s:T_m$ ratio, then looks somewhat as follows: aluminum to 550°F, titanium to 1600°F, and iron to 1500°F. Some difficulties are seen immediately.

In the case of aluminum, it appears that the present service temperature could be increased by about 50°F, which does not appear unrealistic. Current titanium-base alloys are now operating in the temperature range of 900° to 1000°F; thus, it appears that a reasonable future for titanium might be expected. The theory indicates that iron-base alloys may serve up to 1500°F, which is a few hundred degrees in excess of current operating temperatures for iron-base alloys. However, some of the more recently developed austenitic steels are operating at 1400°F; so, it appears that this extrapolation is not too unsafe.

Thus, this is the present and an extrapolated view of what may be expected in the future from the alloy-base materials now being used in high-temperature service. Some aspects of the picture are good, and others leave something to be desired. However, the question immediately is asked, "Is that all that can be expected in the future?" Of course, the answer to this is not definitely known, but there are some things under study that may provide a clue.

Thus, attention focused on higher melting point metals

There are two sides to the alloy picture: one, to design new alloys for higher temperature service at current operating stresses, and two, for higher operating stresses at presently employed temperatures. Regardless of which side of this problem is considered, it appears, from the relation of the service temperature to the melting point of the metal or alloy, that attention should be focused on the higher melting point metals as a method of attacking this problem.

Only those metals are herewith considered whose melting point is in excess of that of iron and consequently above nickel and cobalt. In addition, this discussion is limited to those metals that can be considered from the point of view of economics and availability. If the line is drawn based on these criteria, then the metals for discussion can be limited to the following in order of increasing melting point: titanium, vanadium, zirconium, chromium, columbium, molybdenum, tantalum, and tungsten. Assuming the validity of the earlier relation between the service temperature and the melting temperature ($T_s:T_m = 0.6$), the following might be expected as upper service temperatures for these metals or alloys: titanium 1600°F, vanadium 1700°F, zirconium 1800°F, chromium 1800°F, columbium 2450°F, molybdenum, 2600°F, tantalum 3050°F, and tungsten 3400°F.

Classified by stress:density ratio

The picture looks bright, but there are certain conditions that must be met. Fig. 2 shows the stress to produce 1 pct deformation in 24 hr at 1830°F for these metals in the annealed condition.[3] It is readily observed that there is a sharp change in high-temperature strength between zirconium and chromium, and that between columbium and tantalum little difference in strength is observed. However, tungsten displays excellent resistance to deformation at this temperature. There is, of course, a considerable difference in the density of these elements,

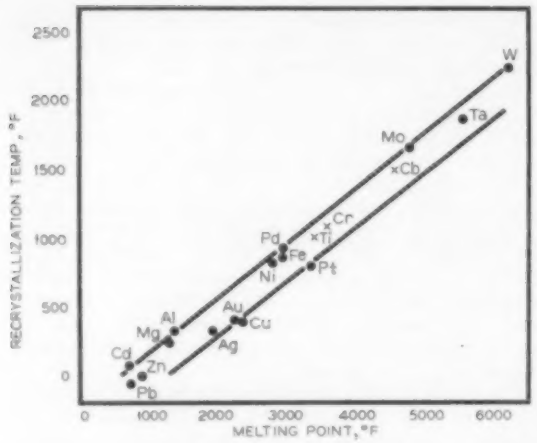


Fig. 1—Approximate relation of recrystallization temperature to melting point of pure metals.

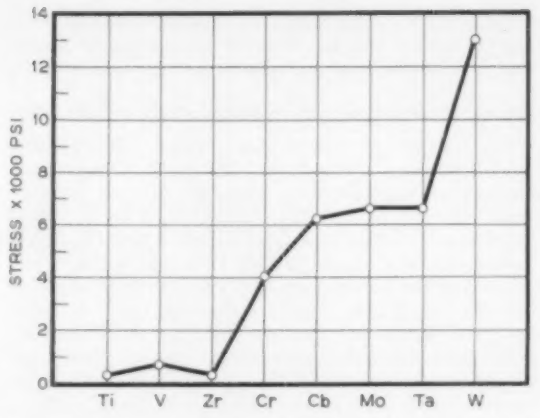


Fig. 2—Stress to produce 1 pct deformation in 24 hr at 1832°F for several metals.

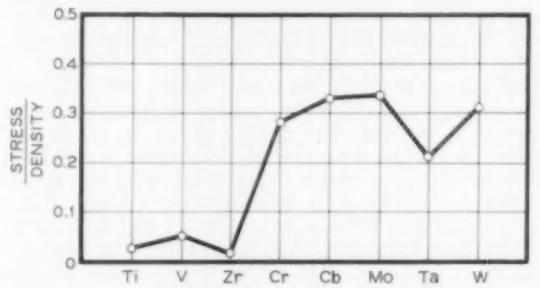


Fig. 3—Stress per unit density to produce 1 pct deformation in 24 hr at 1832°F for several metals.

and if the strength at this temperature is divided by the density a slightly different picture is obtained, Fig. 3.

The lower melting point metals, titanium, vanadium, and zirconium, still have about the same relative positions, but a considerable rearrangement has taken place in the metals of higher melting point. On this basis, the outstanding candidates for service in this temperature range appear to be chromium, columbium, and molybdenum. Although chromium appears very promising on a strength: weight basis, research on chromium and its alloys for many years has not resulted in any commercial alloys for high-temperature service. This probably results because chromium metal itself, unless of extremely high purity, is brittle. In addition, with the exception of a few alloy systems, most chromium alloys develop phases in their microstructure which are brittle and thus render the alloys brittle. Molybdenum would be a very good choice, but the severe oxidation problem of molybdenum has not been overcome even though considerable research has been devoted to this problem. Columbium has a very good strength: weight ratio and is ductile. A problem of oxidation resistance is also present here, but on the basis of current research, a solution may be reached in the near future. Tungsten also appears very promising, but data are limited. Thus, for the service temperature range above 1800°F, columbium and tungsten appear to be the most promising materials.

Fig. 4 shows the stress to produce 1 pct deformation in 24 hr at 1400°F for vanadium, titanium, and zirconium. Vanadium appears to be outstanding, but if considered on a strength: weight ratio basis (Fig. 5) there is very little difference between vanadium and titanium, but zirconium is far less attractive. As far as choosing between vanadium and titanium, perhaps we can apply similar reasoning as used in the case of molybdenum and columbium. Vanadium has poor oxidation resistance and, if exposed at moderate temperatures for a period of time, literally oxidizes away. Also, research on vanadium-base alloys for high-temperature service has been disappointing and no useful alloys have emerged. Titanium, on the other hand, although some oxidation problem exists, shows much better possibilities for future development.

Columbium, tungsten, and titanium offer promise

From the large list of possible alloy base materials for service in the range of 1830°F and up, columbium and tungsten appear to offer the most promise, while for lower temperatures (up to 1560°F) titanium may provide new alloys in the future. The other metals should not be completely excluded, as they may certainly come to the front, even if in only limited applications. However, based on present facts, columbium, tungsten, and titanium should perhaps receive the most attention.

Titanium: From examination of the available information on titanium and titanium alloys for high-temperature service, these conclusions may not be at all unrealistic. In Fig. 6 is shown the 100-hr rupture strength for pure titanium and several titanium alloys as a function of temperature. As shown, selecting a 100-hr rupture strength of 20,000 psi, the ratio of $T_s : T_m$ for pure titanium is about 0.36 which is in good agreement with the value of 0.4 obtained for pure nickel. By alloying titanium with 6 pct Al, the ratio is increased to about 0.44, whereas, if the aluminum content is slightly increased and further alloy additions are made, in this case zirconium and molybdenum, the ratio is increased to about 0.5. Shown on the same curve is the predicted upper service limit for titanium, which is 1600°F, giving a $T_s : T_m$ ratio of 0.6. Thus, by alloying of pure titanium the useful service life has been increased from 800°F to about 1250°F, about half-way to the expected goal. It should be pointed out that this advance has come within a

very short period of time, and only within the last few years has any direct effort been made to design titanium alloys for high-temperature service.

Columbium: In the case of columbium very few high-temperature data are available, as it has been only within the last two years that sufficient quantities of this material have become available. However, several factors combine to make columbium attractive for high-temperature structural use. These include high melting temperature, good elevated temperature strength, and medium density. Unfortunately, columbium must be protected from oxidation when used at high temperature in air, and at present it is relatively expensive. Although the room temperature tensile strength of columbium is not exceptional, varying from 50,000 to 100,000 psi, its loss of strength with increasing temperature is quite gradual. Also, for nuclear-reactor use, columbium has a decided advantage over molybdenum because of its lower absorption cross-section for thermal neutrons.

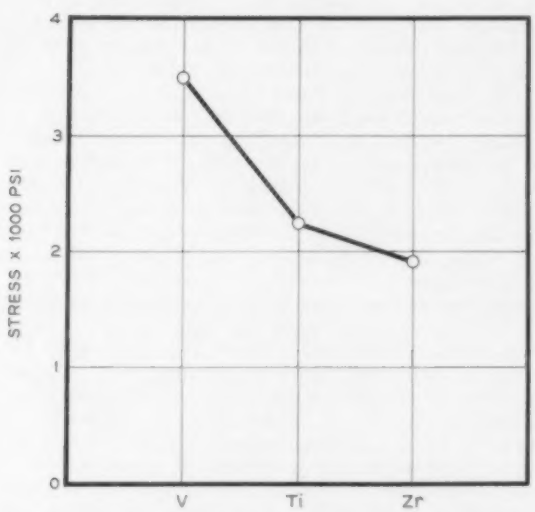


Fig. 4—Stress to produce 1 pct deformation in 24 hr at 1500°F for vanadium, titanium, and zirconium.

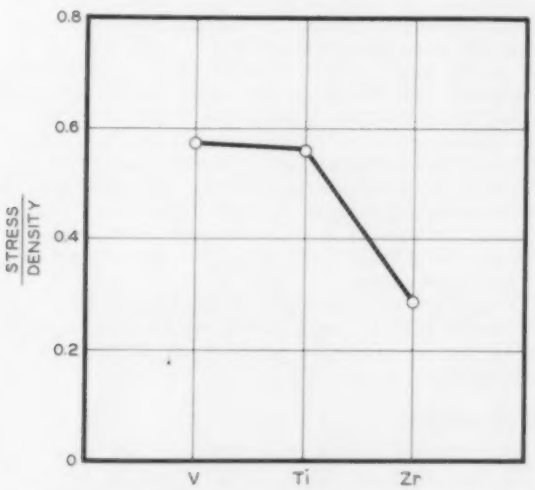


Fig. 5—Stress per unit density to produce 1 pct deformation in 24 hr at 1500°F for vanadium, titanium, and zirconium.

Fig. 7 shows some of the limited data available on columbium and columbium alloys. From the stress-rupture data shown, the 100-hr rupture strength of cold-worked columbium is about 16,000 psi at 1800°F. This would give a 100-hr life at 20,000 psi at approximately 1750°F and result in a $T_s:T_m$ ratio for columbium of about 0.42, which is again in good agreement with the prediction established with nickel. It is interesting to note the strength of columbium at 1800°F as compared to Haynes Stellite No. 31 alloy, which is the best superalloy for this temperature range of operation. The addition of titanium, although it improves the workability of columbium, has a deleterious effect on the high-temperature behavior. It is too early to definitely establish the upper service temperature for columbium-base alloys, but the preliminary results seem to indicate the future looks bright, and the earlier goal of 2450°F may be a reality of tomorrow.

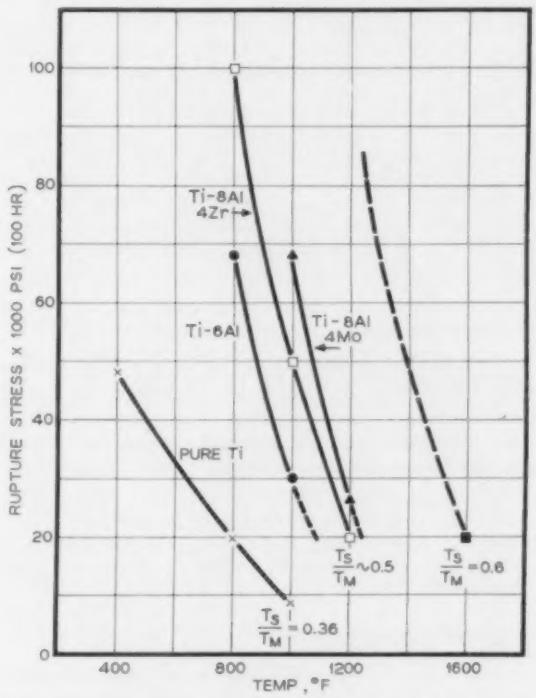


Fig. 6—Stress-temperature curves for rupture in 100 hr for titanium and titanium alloys.

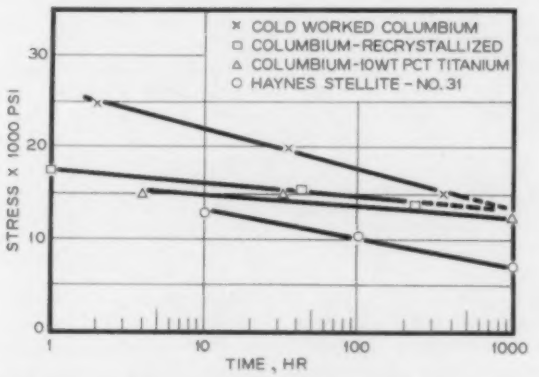


Fig. 7—Stress-rupture curves for columbium and a columbium-titanium alloy at 1800°F.

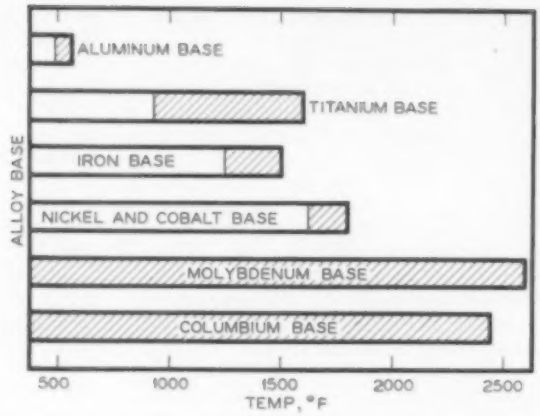


Fig. 8—Extrapolated maximum service temperature for several high-temperature alloy base materials for 100-hr rupture life at 20,000 psi.

High-temperature alloys of tomorrow

This is some indication of what tomorrow may hold in terms of metals and alloys. This picture has been oversimplified; production and fabrication costs have not been considered, and many important properties of metals have not been taken into account, however, the final revised picture is shown in Fig. 8. The conclusions drawn, with respect to the high-temperature alloys of the future, are:

1) Titanium-base alloys may be expected which will be useful in the range to 1600°F;

2) Columbium-base alloys well may be developed which will be useful at temperatures in the range to 2500°F;

3) Molybdenum-base alloys should be found suitable for service in the range to 2600°F; and

4) Tungsten-base alloys will be developed with useful strength up to the range to 3400°F.

Much research is required to reach these goals, but just as research has been fruitful in the past, there is confidence that it will be in the future.

Atomic power

Most metallurgists are aware of the increased use of **zirconium** in the atomic power industry, and some zirconium production figures can be used to illustrate the activity in this industry. In 1948, production was about 100 lb per year. In 1956, sponge capacity was approximately 235 tons. The 1959 capacity for making zirconium sponge is expected to reach something over 3000 tons. Zirconium has been used in heterogeneous reactors, such as those installed at Shippingport, Pa., and those in the U.S.S. Nautilus. More advanced power reactors of the fast-breeder type are expected to use materials other than zirconium in significant quantities.

Facilities for the generation of electric power from atomic energy are being installed today. By 1965, about 4 million kw of capacity are expected. Estimates for the installed capacity by 1975 vary between about 50 and 80 million kw, and for 1980—between about 100 and 200 million kw. **Columbium and vanadium** are both being studied for application in reactors of advanced design. It has been estimated that nuclear reactor cores may contain over 100 lb of columbium, both as a canning material and alloyed with uranium. Too little is known about different reactor types proposed, or under test, to be able to make very definite predictions about the future of columbium or vanadium, but it appears that columbium and/or vanadium have a good chance of providing the properties necessary for important applications in this complex, new industry.

Production and availability

To produce the newer metals and alloys and to fulfill the needs of the new industries, entirely new technologies have had to be introduced into the metallurgical field. Prior to this new era, practically all the metals, with the possible exception of aluminum, were won from their ores through a high-temperature smelting technique involving large furnaces with arcs or flames, complicated oxidizing and reducing steps and such operating problems as slag control and deoxidation. Manufacture of the new metals, however, involves a substantial amount of chemical processing. New techniques include fluidized beds, purification and extraction columns, piping and valving of liquid metals, and arc melting ingots of alloys in water-cooled molds.

Although many of these elements have been considered rare in the pure state, they exist in nature in relative abundance. Ttitanium, the prime example of the new metallurgy, is extremely abundant and is actually ninth in degree of abundance in the earth's crust. It exists in several distinct ores including ilmenite, rutile and euxenite, most of which are quite readily adaptable to further processing. Its abundance is a strong encouragement to further promotion of the metal as an engineering metal to take its place along with the other large tonnage base alloys, such as iron, aluminum, and copper.

Columbium and tantalum do not exist in such world-wide abundance, but they are not scarce metals. Columbium is about one-third as abundant as copper, and equally as abundant as cobalt. Tantalum exists in about the same percentage as molybdenum and is much more abundant than the precious metals such as silver, gold and platinum. Tantalum and columbium nearly always occur together, but there are a reasonable number of tantalite deposits, particularly in Brazil, where the ratio of tantalum to columbium is quite high. Columbite-tantalite deposits occur principally in Nigeria, Belgian Congo, and Southern Rhodesia and are of sufficient quality to justify processing by conventional methods. Columbium and tantalum are also associated with many tin deposits in Malaya and Nigeria, and hence are essentially by-products. In addition, columbium exists in small percentages in very large pyrochlore deposits in several large areas in the world and is also associated with many bauxites. Thus, it is believed that there is much more columbium available in the world than is required for any demand that is now envisioned.

Vanadium is relatively abundant and has been produced in fairly large quantities for its own characteristics as well as a by-product of the uranium operations.

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REFRACTORY METALS: TUNGSTEN, TANTALUM, COLUMBIUM, AND RHENIUM

An analysis of the status and prognosis of these four refractory metals with respect to high-temperature applications.

by J. W. Pugh

IT seems likely that the greatest impetus for the development of refractory metals and alloys will come from the demand for jet, rocket, or nuclear engines, although many other groups, such as the lamp and the electronic tube industries, have a considerable stake in refractory metals development.

Thus far, most of the effort has been devoted to the search for a satisfactory molybdenum alloy with a coating. To a large extent other refractory metals have been overlooked, because of their cost, lack of availability, density, and fabrication disadvantages. However, some of these other metals are stronger than molydenum, some are more ductile, and all, except rhenium, are more oxidation resistant at high temperatures.

All four metals can be arc-melted, sintered, forged, swaged, and rolled. It is likely that all may also be extruded, if techniques similar to that used for molybdenum are developed. But fabrication frequently does require some care in preparation or schedule, for example, interstitial impurities, such as oxygen, are deleterious to the formability of arc-melted columbium,[1] rhenium cannot be cold worked more than about 30 pct without intermediate annealing,[2] and tungsten has a relatively high brittle-ductile transition temperature, and care must be taken to maintain the fabrication temperature above the transition temperature which is characteristic of the structure being deformed.[3]

Properties compared

There are a number of properties other than the obvious strength, ductility, and oxidation resistance which determine a refractory metal's aptitude for high-temperature structural applications. A summary of these properties for tungsten, rhenium, tantalum, and columbium is made in Table I. Molybdenum is included in this tabulation for the purpose of comparison. The metals are arranged from left to right in diminishing order of their melting points; this order is almost the same as their density. Refractory metals are comparatively stiff, with the exceptions of tantalum and columbium whose Young's Moduli are less than that of steel. Rhenium is the only refractory metal which is not body-centered-cubic, and its hexagonal, close-packed structure may account for its unique mechanical properties.

The comparison of rescrystallization temperatures is not precise, since it relates metals of differing purity and prior deformation. In general, these tem-

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Table I. Properties of Refractory Metals[1]

Property	W	Re	Ta	Mo	Cb
Melting Point, °C	3410 (4)	3180 (2)	2996 (4)	2623 (4)	2415 (4)
Density, g per cu cm	19.3 (4)	21.0 (2)	16.6 (4)	10.2 (4)	8.57 (4)
Young's Modulus, 10^6 psi	51 (3)	67 (2)	27 (4)	47 (5)	15.1 (6)
Structure	BCC	HCP	BCC	BCC	BCC
Recrystn. Temp., °C (1 hr)	1565 (7)	1400 (2)	1275 (8)	1130 (9)	1050 (4)
Linear Therm. Exp., coeff. 10^{-6} per °C	4.45 (3)	6.7 (2)	5.9 (10)	5.6 (11)	7.1 (4)
Therm. Cond., cal per cu cm, °C, sec	0.48 (4)	0.17 (2)	0.13 (4)	0.35 (4)	0.125 (12)
Vapor Pressure, μ at 2225°C	1.33×10^{-4} (13)	1.18×10^{-3} (2)	7.94×10^{-4} (13)	0.50 (13)	1.66×10^{-2} (13)
Cost for powder, $ per lb	4.75	680.00	43.00	3.40	100.00

[1] Note: Numbers appearing in parentheses are reference numbers.

peratures are based on 1 hr annealing treatments after more than 40 pct cold reduction. There is a distinct relationship between the recrystallization temperatures and the melting points.

Thermal coefficient of expansion and thermal conductivity are, to some extent, indicative of thermal shock resistance. The values in Table I were obtained at or near room temperature. Tungsten has the most favorable combination of these properties.

Vapor pressure is of particular interest to the lamp and electronic tube manufacturers, and it is easy to see why tungsten and tantalum are favorite refractory metals in this industry.

The cost figures in this table vary considerably with vendor and purity. Some change significantly with time, particularly columbium which is rapidly becoming cheaper. On the basis of cost, tungsten is currently most attractive.

Tensile parameters studied

Mechanical strength is the property of primary importance to the high-temperature application of refractory metals. It is frequently possible to gain considerable understanding of mechanical behavior characteristics by studying short-time tensile parameters in detail. In this respect, it appears possible to classify body-centered-cubic refractory metals according to their position in the periodic table. Those which occupy Group V-a tend to have relatively high solubility for interstitials and no brittle-ductile transition temperature; they are greatly affected by strain aging. Those found in Group VI-a have low solubility for interstitials and a definite structure-sensitive transition temperature; they are considerably less affected by strain aging.

Tantalum is typical of Group V-a metals; tensile parameters for tantalum as a function of temperature are presented in Fig. 1.[14] Here the strain hardening is represented as the slope (m) of the logarithmic stress-strain curves. It is defined by the relationship:

$$m = \left.\frac{\partial \log \sigma}{\partial \log \epsilon}\right|_{\dot{\epsilon}, T},$$

where m is called the strain hardening exponent, σ is the flow stress, ϵ is the strain, and T is temperature.

A similar relationship is used to evaluate rate sensitivity,

$$n = \left.\frac{\log \dfrac{\sigma_1}{\sigma_2}}{\log \dfrac{\dot{\epsilon}_1}{\dot{\epsilon}_2}}\right|_{\epsilon, T},$$

where n is called the strain rate sensitivity; σ_1 is the flow stress at one testing rate, and σ_2 is the flow stress at a faster rate; $\dot{\epsilon}_1$ is the faster rate, and $\dot{\epsilon}_2$ is the slower rate. There are several significant features of the temperature dependence of these parameters, for example, strength is strongly dependent on temperature in the low temperature range. The minima in strain-rate sensitivity and elongation and the maxima in the strain hardening and strength relationships are all indicative of strain aging. No brittleness is encountered at temperatures as low as liquid nitrogen temperature, although elongation does drop to a low value at this temperature. Columbium and vanadium also belong to Group V-a and can be expected to have similar mechanical behavior. Recent data for vanadium corroborate this.[16]

The tensile properties of tungsten, illustrated in Fig. 2, are typical of Group VI-a.[16] In this case, anomalies in the temperature dependence of these parameters, which might indicate strain aging, are less evident. The peak in the strength data of Bechtold and Shewmon[17] is the only clear-cut evidence which might indicate strain aging. Note that there is very definitely a temperature (approximately 500°K) below which brittleness is encountered. This temperature depends, of course, on the structure of the metal and the test conditions. Other body centered cubic metals in Group VI-a which qualify for classification with tungsten are chromium[18] and molybdenum.[19]

In Fig. 3, the tensile strengths of all the four refractory metals under consideration are plotted as a function of testing temperature. Rhenium[2] is obviously superior over the whole temperature range. A considerable measure of this superiority is due to a remarkable ability to strain harden. This property at room temperature in terms of the popular logarithmic stress strain curve slope is 0.372, compared to a value of approximately 0.2 for molybdenum and tungsten. A more realistic basis for comparing materials of significantly different strengths is the slope of the unmodified true stress strain curve. On this basis, rhenium strain hardens at about 3.5 times the rate of tungsten or molybdenum. Exceptionally high strength and high strain

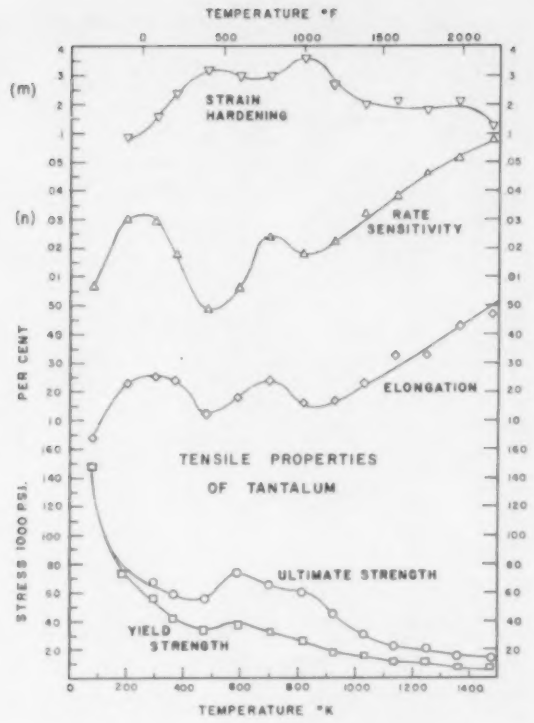

Fig. 1—Tensile parameters for tantalum—typical of Group V-a metals—as a function of temperature.

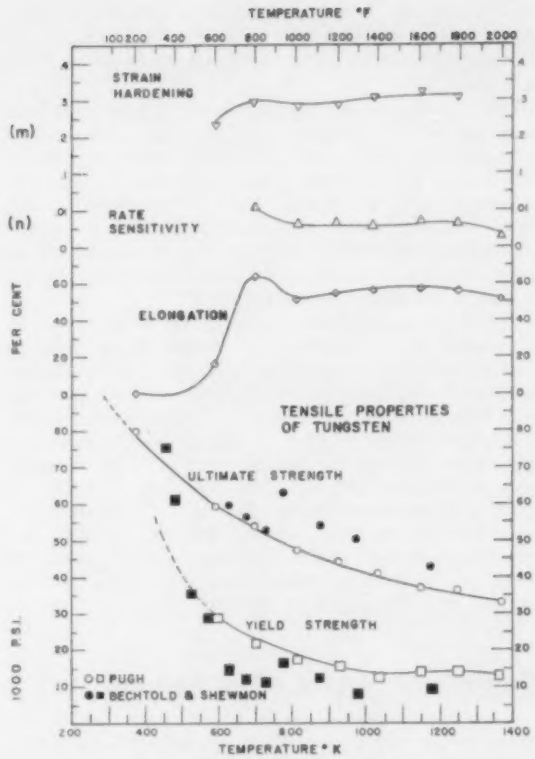

Fig. 2—Tensile parameters for tungsten—typical of Group VI-a metals—as a function of temperature.

hardening in rhenium probably result from the combination of few available slip systems, a c:a ratio (1.615) which precludes deformation by twinning in tension, and the same disinclination to thermally activated recovery at low and moderate temperatures which is common to all refractory metals. The discontinuity in the rhenium curve corresponds to the recrystallization range.

The strain aging peak in the tantalum data has been eliminated here to avoid confusion, and the relationship of columbium[1,4] is dotted because reliable data at intermediate temperatures are lacking. Single point data for some other metals is plotted at high temperatures for the purpose of comparison. Note that the strength level for all these metals, except tantalum which is slightly out of line, appears to be in the order of the respective melting points.

A convincing demonstration of the relationship between melting point and the strength level has been made for the Group VI-a metals. For this purpose they were each tested under identical conditions but compared at the same fraction of their melting points. Fig. 4 shows the strength of these metals as a function of homologous temperature or test temperature in per cent of the absolute melting point. This is a striking indication of the correlation between strength and melting point and illustrates the advantage of the more refractory materials.

Creep rupture strength

Another significant parameter is that of creep rupture strength. Fig. 5 shows rupture strength as a function of temperature for 10 hr life. Molybdenum[19] is compared with tungsten,[16] rhenium,[7] and columbium;[1] no data for tantalum were found. Rhenium shows less superiority over tungsten at the high temperatures. Molybdenum and columbium both appear considerably weaker. A comparison of stress-rupture properties based on the Larson-Miller parameter[20] shows that rhenium is again most outstanding at lower temperatures and for shorter times. The order of superiority for body-centered-cubic metals again appears to be related to their melting points.

Oxidation behavior

The strength of these refractory metals at high temperatures is a remarkable characteristic; their rapid destruction by oxidation at these same high temperatures is another. Rhenium is similar to molybdenum in its oxidation behavior.[7] It oxidizes catastrophically above about 600°C, because of the formation of rhenium heptoxide (Re_2O_7) which melts at 279°C and boils at 363°C. Tantalum and columbium appear to be similar to each other in their oxidation behavior, but the most definitive work at high temperatures has been done on columbium.[21]

Oxidation of columbium in air becomes serious at about 400°C. The oxide formed is one of three modifications of columbium pentoxide (Cb_2O_5). The modification formed (*T*, *M*, or *H*) depends on exposure temperature, and each modification gives rise to a different activation energy. The melting point of columbium pentoxide is 1440°C, and no significant loss from vaporization is encountered as high as 1370°C. This information puts columbium in a considerably more favorable position than molybdenum or rhenium. However, the rapid diffusion

rate of oxygen in columbium is a detriment. Embrittlement, due to high temperature exposure in air, may prove to be a more serious hazard than oxidation.

The high temperature scaling of tungsten has also received some attention.[22] It becomes serious only above about 600°C. The scale apparently consists of two layers which differ from each other in the degree to which they deviate from stoichiometric ratio WO_3. Scale growth is determined by two processes: the diffusion of oxygen and the volatility of the scale. Up to about 1350°C the oxygen diffusion is said to be more important.

Fig. 6 shows the relative oxidation attack for 1 hr exposures as a function of temperature for the four refractory metals. Rhenium[5] has the worst performance and tungsten[22] the best, but all are intolerable for high temperature applications. No satisfactory remedy for this problem has been found, but there is hope for the development of satisfactory protective coatings and for the invention of oxidation-resistant alloys. Perhaps, the most probable remedy is the combination of coatings and resistant alloys. It is expected that, in this case, defects in the coating will be compensated, to some extent, by the superior scaling resistance of the alloy.

Oxidation-resistant alloys and coatings

Very little information on the subject of oxidation resistance or protection for these metals is yet available; however, there are two developments worthy of mention. The first is the extension of the molybdenum disilicide coating technique to tungsten.[24] The tungsten disilicide coating can be readily produced by the catalytic reduction of silicon tetrachloride and is said to give prolonged protection up to 1500°C. This is approximately 100°C higher than the effective range of molybdenum disilicide. However, such coatings appear to be even more brittle than the molybdenum disilicide coatings at room temperature.

The other development is that alloys of columbium and titanium, which contain ternary additions of molybdenum, chromium, and vanadium, have shown improved oxidation resistance by a factor of approximately 10 over unalloyed columbium.[25] This is not nearly good enough but, perhaps, it does indicate some reason for optimism.

Very little effort has been expended thus far on the development of tungsten, tantalum, rhenium, and columbium alloys. There are some new efforts in this area, and these may indicate an acceleration of interest. Among the positive results of these efforts is the discovery that the addition of rhenium to tungsten causes room-temperature ductility,[26] and the revelation that adding titanium to columbium promotes fabricability.[1] But these are only the first explorations into a relatively unknown metallurgical frontier.

Conclusions

It may be appropriate to speculate a bit about the future of refractory alloy design. There are some relatively good commercial tungsten materials which are used today in the lamp industry. Most of these resulted from literally hundreds of experiments in which everything imaginable was attempted. Some of these experiments took place 50

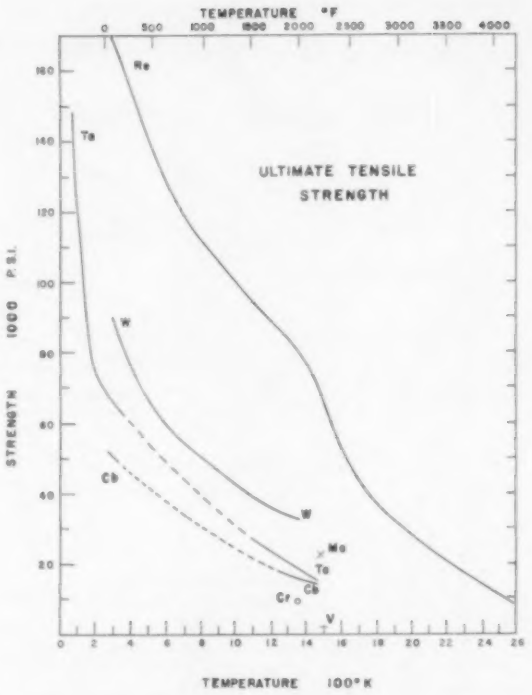

Fig. 3—Tensile strengths of refractory metals as a function of temperature.

years ago. One of the tungsten alloys which resulted from this early melange of experimentation was thoriated tungsten.[8] One of the desirable properties of this material is its exceptionally high strength. In general, the microstructure of this thoriated tungsten is one of very fine grain size with finely dispersed particles of relatively insoluble ThO_2 which tend to be segregated at the grain boundaries. Now the interesting thing about all this is that this is almost precisely the microstructure prescribed recently by Mott[27] as ideal from a theoretical point of view.

There is a message in this coincidence. It is this: the refractory alloy designer of today has an advantage over those who designed current commer-

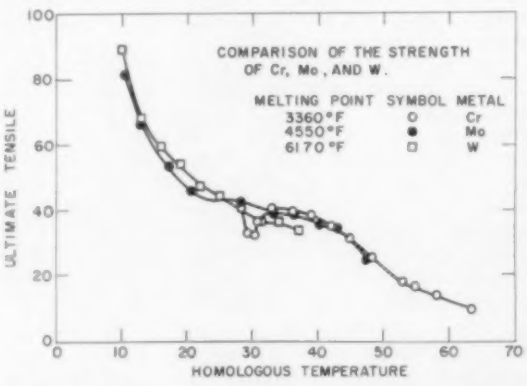

Fig. 4—Strength of refractory metals as a function of homologous temperature, or test temperature, in per cent of the absolute melting point.

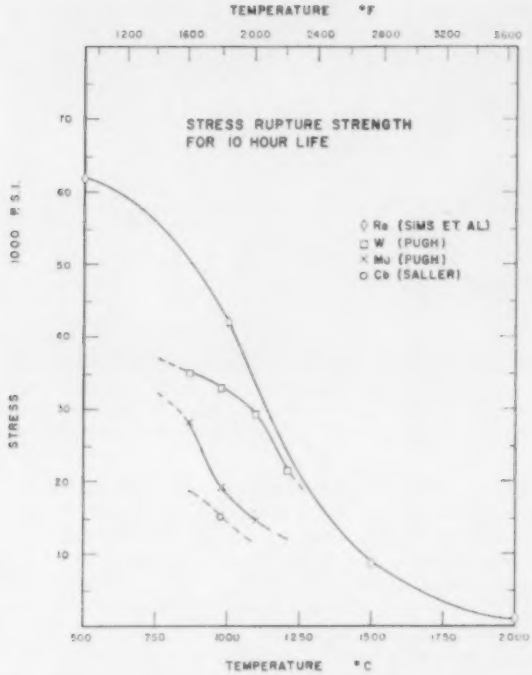

Fig. 5—Rupture strength as a function of temperature for 10 hr life.

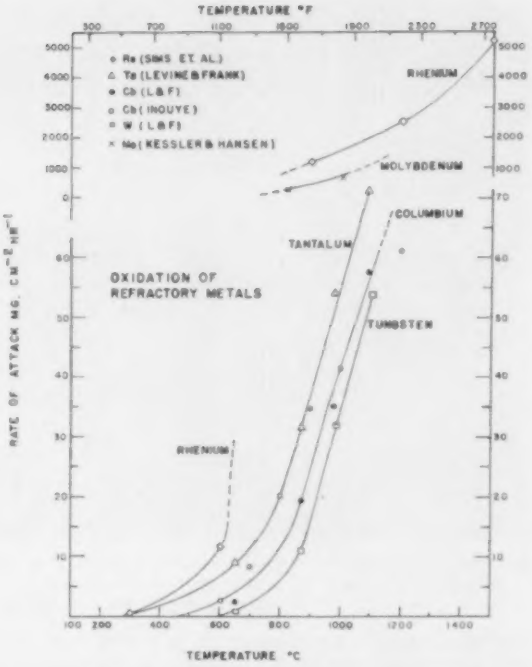

Fig. 6—Relative oxidation attack for 1 hr exposures as a function of temperature.

cial high-temperature materials, because he can approach the task with more knowledge about the fundamental nature of alloying. He has new theories on dispersion hardening by Mott,[28] and by Fisher, Hart, and Pry[29] to guide him. Moreover, the ideas of Fisher[30] and of Cottrell[31,32] on solution hardening should prove helpful. Also of considerable importance are the recent experiments relating polygonization and subgrain growth to mechanical strength.[33,34,35] Substructure hardening is particularly well suited to refractory metals because, in general, their recrystallization temperatures are high relative to application temperatures.

Perhaps the assistance available from these new basic concepts will compensate in some measure for the additional problems of brittleness and oxidation attack which face the designer of refractory alloys.

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A COLUMBIUM PRIMER

Interest in metallic columbium and in columbium-base alloys is increasing rapidly, since they have an excellent potential for use at high temperatures in atomic energy applications and in missile or jet-engine applications.

by Chester T. Sims

THE attraction for columbium is due to both economic and technical reasons. During and after the Korean emergency, columbium was in short supply. However, in May, 1952, the US General Services Administration announced a program to stimulate production by authorizing stockpiling of the metal at several key refineries and paying an incentive bonus. By May, 1955, 15 million lb of concentrate had been stockpiled, and new sources of the metal were being rapidly uncovered. Later the Government buying program was terminated, and ample supplies of columbium now seem assured.

Occurrence and economics

The most common natural minerals containing columbium are *columbite* and *pyrochlore*. Columbite is a complex ferrous or manganous columbite-tantalate, (Fe, Mn) $[(Cb, Ta)O_3]_2$, in which the columbium dominates. When tantalum dominates, the mineral is called tantalite. Columbite is usually found in granite pegmatite rocks or aluvium produced from pegmatites. Pyrochlore, which occurs in alkalic rocks, is a fluocolumbate of calcium and sodium, and is usually associated with rare-earth minerals. Columbium-bearing pyrochlore ores contain about 0.2 to 2.0 pct Cb_2O_5. Columbite ores found overseas possess average columbium-to-tantalum ratios of approximately 3:1, although North American pyrochlores, which are receiving considerable attention, have a 50 or 100:1 ratio. It is easier to separate columbium from columbites than from pyrochlores and, up until the present, most columbium metal has been separated from columbite-type ores.

The US consumes a major portion of the world columbium supplies,[1] much of which is imported from Nigeria or the Belgium Congo.[2] The African ores are both of the columbite and pyrochlore type, although most of the metal actually separated comes from the columbites as a by-product of tin-mining operations. Large bodies of pyrochlore ore have recently been uncovered in eastern Canada.[3] Other important new sources of columbium have recently been found in British Guiana, Malaya, and South America. Table I summarizes the columbium concentrates supply picture as it appeared in mid-1957. Recent estimates[2] place the world reserves at about 1.5 million lb of contained columbium, which is probably ample metal to supply expected demand for many years, even if the use of columbium increases sharply.

US marketers of pure columbium or columbium carbides are the Fansteel Metallurgical Corp., Kennametal, Inc., Electro Metallurgical Co., div. of Union Carbide Corp., Wah Chang Chemical Corp., Stauffer Chemical Co., Shieldalloy Corp., and Kawecki Chemical Co. Fansteel is the only domestic producer currently selling pure columbium in fabricated form; but columbium mill products may also be purchased from overseas.

A large price gap exists between the price of pure fabricated metal and the technical grades. This large differential is due to purification costs and to present small-quantity production. Since columbium is one of the more fabricable refractory metals, fabrication is not expensive. The purification problem is under intensive study by several domestic producers. Ultimately, the technical difficulties are expected to be overcome, and a market price for high-purity columbium of $10 to $15 per lb or less is visualized.[4] Technical-grade columbium pellets are already available in this price range.

Extraction and preparation

Since columbium often occurs closely associated with tantalum in nature, the two metals are usually separated unless columbium-tantalum alloys or ferro-columbium-tantalum, used in industry for stabilization of stainless steels, is desired. Concentrated ores, with or without tantalum, intended for this purpose are converted into the iron complex by carbon-arc reduction and sold as ferroalloys. Ores intended for processing into pure metal undergo a much more complicated chemical processing before being made ready for consolidation.

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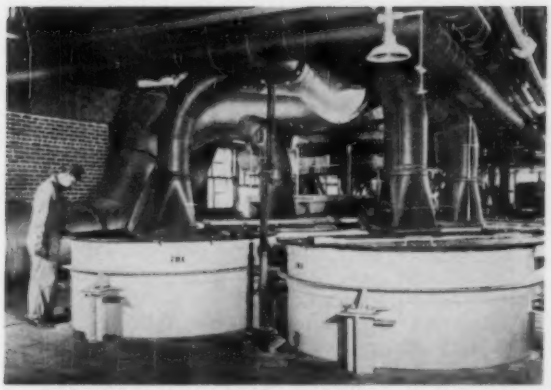

Fig. 1—Separation of tantalum from columbium. In the treatment of columbite ores, tantalum is crystallized out as a complex salt, while columbium is retained in solution. Courtesy, Fansteel Metallurgical Corp.

If alkali extraction is used,[5] the ore is fused with molten caustic, treated with water, hydrochloric and hydrofluoric acid, and columbium separated from tantalum as a potassium-columbium-oxyfluoride complex, Fig. 1. The impure, tantalum-free columbium concentrates are treated further until finally columbium pentoxide (Cb_2O_5) is prepared from potassium columbate ($KCbO_4$). The ore may also be fused with sodium pyrosulfate, treated in a somewhat analogous fashion to that outlined above, and the columbium-rich portion finally removed from the tantalum by concentrated sulfuric acid extraction. Preparation of raw metal is accomplished by reduction of columbium pentoxide with columbium carbide during vacuum sintering.

Other methods of preparation of columbium metal are also known.[6] Electrolysis may have an advantage, and a promising process involving electrolysis of K_2CbF_7-NaCl mixtures has recently been described. In this process, K_2CbF_7, which is available as an intermediate product in ore concentration, is electrolyzed to high-purity, ductile columbium metal at about 1500°F while mixed with amounts of from 10 to 14 pct NaCl.

Another production process, which has been proposed, is hydrogen reduction of columbium chloride; $CbCl_5$ could be prepared from a low-tantalum columbite or a pyrochlore ore and then reduced to metal. In the past, high-purity columbium has also been prepared by thermal decomposition of a volatile columbium halide, such as the pentachloride. This method could be expected to produce somewhat higher purity columbium than direct hydrogen reduction.

Two principal methods of consolidating columbium metal for fabrication are possible. Most of the columbium mill products being sold today are prepared by sintering in high vacuum as discussed below. However, since purified columbium is very easy to work, commercial production of ingots consolidated by consumable-electrode arc melting (as now being done with molybdenum) is promising. Considerable experimental work is under way, but obtaining sufficient purification during consolidation, which is critical with columbium, remains a problem and must be solved to permit economical processing by arc melting.

Vacuum purification

Vacuum purification is the best known method of preparing columbium metal. Commercial[8] vacuum purification is accomplished in two major steps. First, pressed bars of mixed columbium oxide and carbide are sintered at 3100° to 3450°F in high vacuum (Fig. 2) where the two chemicals react to form columbium metal and carbon monoxide. The latter, of course, is pumped off by the vacuum system. The columbium bar, too impure to be fabricable, is broken up, re-pressed, and re-sintered at 4200°F. Sometimes, a third sintering is given. The ductile bars, purified by the extra sintering, are then fabricated to mill products.

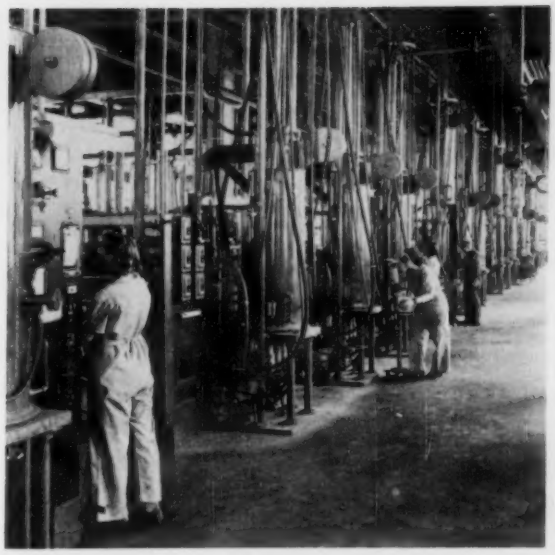

Fig. 2—Commercial vacuum sintering of columbium. Bars are heated electrically in high vacuum in these vertical chambers. Courtesy, Fansteel Metallurgical Corp.

Recently, some of these vacuum reactions were studied in detail.[7] It was found that impure columbium powder, containing up to 2 wt pct O_2, 0.5 wt pct C, and 0.3 wt pct N_2, could be pressed into bars and sintered to a fabricable product in high vacuum at 4000°F or above. Carbon and oxygen were removed as carbon monoxide at about 2500°F. Excess carbon is stable and remains in the bar, so that for highest purity it is advisable to have a slight excess of oxygen. The remaining oxygen can then be removed by a second reaction, vaporization of CbO, which occurs at about 3300°F.

Nitrogen is removed by simple evaporation of nitrogen gas at about 3500°F, although the reaction is very slow. Columbium containing 0.25 wt pct or less O_2, 0.08 wt pct or less N_2, and about 0.05 wt pct or less C can be readily fabricated to sheet or rod.

Fabrication

Pure columbium is one of the most workable of all refractory metals. Commercially fabricated columbium can be rolled, forged, swaged, drawn, spun, and stamped at room temperature.[5] However, it tends to gall or seize on the working tools so that proper lubrication by use of light oils is recommended. Working operations can be conducted to a considerable degree, since the tendency of columbium to work harden is very low.

Vacuum-sintered columbium bar can be rolled or swaged directly to sheet or rod at room temperature. However, for best results, it is recommended

that about 10 pct reduction in cross-sectional area be taken, the bar resintered to heal voids, and then fabrication to the desired size continued.

Arc-melted columbium or columbium-base alloys can also be handled easily, although a few precautions are necessary. One procedure used experimentally is to machine parallel top and bottom surfaces on the as-cast columbium or columbium alloy ingots prior to forging to reduce cracking while the large grain as-cast structure is being broken up. Press forging followed by rolling can then be done cold. Another procedure is to use a V-block under the ingot during forging. This provides sufficient triaxial restraint to materially assist breaking up of the as-cast structure.

With care, columbium can be heated to assist fabrication.[8] Although columbium should not be heated in air for a long time, since inward diffusion of oxygen will cause embrittlement, rapid air heating up to 2200°F prior to working cast material is acceptable. This will assist breaking up of the large-grain as-cast structure, and the oxygen content will not increase significantly. Heating to working temperatures of about 2000°F in helium has also been done successfully with subsequent fabrication occurring in air. Heating in a reducing atmosphere may also be possible but only with care, since columbium converts entirely to a hydride on slow cooling in hydrogen from 2000°F. In this case, it may be possible to heat columbium in hydrogen above the range for hydride formation, then remove and fabricate in air. If fairly rapid cooling occurs, inward diffusion of oxygen would be kept to a minimum and successful working accomplished. This procedure has been practiced for tantalum[9] and might result in less metal loss during fabrication for columbium. For simple annealing, a high vacuum is recommended. Columbium can be joined by inert-gas arc welding.

Properties

Columbium is similar to molydenum in many respects. The two metals are in adjacent positions in the periodic system, crystal structure is the same, and melting point, vapor pressure, and density are fairly similar. This makes them of nearly equal interest for many applications.

However, the two metals also differ in some important respects. Columbium has a low thermal-neutron cross section, while that of molydenum is considerably higher. As discussed below, this makes columbium of interest as an alloy base for reactor construction materials. Columbium has lower electrical and thermal conductivity than molydenum, which puts columbium at a disadvantage for many applications where high heat transfer is needed, such as in a reactor or in aircraft engine parts. The low modulus of elasticity of columbium is reflected in the ease of fabricability and the low strength at room temperature compared to molydenum.

Columbium has very interesting mechanical properties. Although its room-temperature strength is moderate (see Table II), strength is retained with very little decrease as temperature increases. As shown in Fig 3, the ultimate tensile strength of columbium at 1200°F is approximately the same as at 200°F, and is very little lower than at room temperature. Since columbium also has a low cross section, this excellent strength retention with increasing temperature has made it of prime consideration for use in nuclear reactors as a fuel sheet matrix material.

Stress-rupture data has been obtained for columbium in the region of 1600° to 2000°F, a critical temperature region when design of gas turbine and other heat engines is considered. Fig. 3 shows that hot-cold-worked molybdenum has somewhat superior stress-rupture properties over this range, although this superiority is only slight above 2000°F. It should be realized, however, that molybdenum alloys with 100-hr stress-rupture strengths of the order of 100,000 psi at 1800°F are freely predicted,[11] while development of high-strength columbium-base alloys is in its infancy.

Alloys

A summary of the known alloying behavior of columbium is given in Table III. As would be expected, BCC columbium generally forms a complete series of solid solutions with other principal refractory alloying elements, most of which are also BCC, including molybdenum, tungsten, tantalum, β-titanium, β-zirconium, and β-thorium.

Since columbium itself is readily hot and cold workable, fabrication of binary or ternary solid-solution alloys with other high-melting metals is likely to be less difficult than with molybdenum.

Table I. Approximate US[a] and Canadian Production and Imports and World Reserves of Columbium

Source of Concentrate (by Continents)	US Shipments or Imports, Lb of Cb 1956	Cb_2O_5 Content, As Shipped, Pct	Type of Ore	Estimated Reserves Tons of Cb
North America (US and Canada)	(140,000)[b]	—	Columbites, pyrochlores, euxinite and bauxite	320,000
South America (Argentina, Bolivia, Brazil, British Guiana)	20,300	49 to 50	Pyrochlores	1,700,000[c]
Europe (Belgium-Luxembourg, West Germany, Norway, Portugal, U.K., Spain)	563,000	37 to 41	Pyrochlores and koppite	350,000
Asia (Malaya, Aden)	523,000	55		—
Africa (Belgian Congo, Kenya, Madagascar, Mozambique, Nigeria, Rhodesia, Nyasaland, Uganda, Union of South Africa, British East and West Africa, French Equatorial Africa)	4,430,000	40 to 56	Columbites and pyrochlores	890,000
Australia (Oceania)	—	42	Columbites	—
Totals	5,600,000	51		1,560,000

[a] United States imports about 85 percent of world columbium production.
[b] Parenthesized figure is combined columbite-tantalite.
[c] Unconfirmed estimate. Not included in total.

This has been confirmed by Stacy[6,15] and co-workers who forged and rolled arc-cast Cb-5Zr, -5Mo, -5Cr, -5V, -5W, and -5Ta alloys at room temperature. Further, arc-cast columbium-titanium alloys are fabricable up to at least 20 wt pct Ti, and Cb-10Ti is more fabricable than pure columbium. This is probably at least partially due to a reaction between titanium and interstitial oxygen present in the columbium to form TiO_2. Ternary additions of titanium assist the workability of the alloys listed above and also allow working of alloys containing iron, columbium, and nickel. Alloys of this sort prepared by powder metallurgy are also workable.[15]

Data on the strengthening of columbium by alloying has been slow to be published in sufficient volume to allow a firm picture to be drawn. Tottle[12] has given some room temperature data which shows that additions of up to 10 pct Mo, Ta, and W, 5 pct Ti, and 2 pct V produce significant strengthening up to slightly more than double the strength of the columbium starting material. This strengthening is consistent with good ductility. However, 0.3 pct O_2 will triple the strength of columbium at room temperature, with a significant degree of ductility still retained.

Hardnesses have been obtained at room temperature and at 1600°F on the series of binary alloys discussed above and on alloys containing titanium as a ternary addition.[6] Five and 10 pct Zr alloys showed excellent retention of hot hardness from room temperature to 1600°F. Conversely, titanium ternary additions produced softening. Since no tensile data were obtained, a comparison can not readily be made with molybdenum. However, the hardness values for these alloys are very roughly two and one half times the ultimate tensile strength in thousands of pounds per sq in., and on this basis, the zirconium alloys have an ultimate tensile strength of approximately 90,000 to 100,000 psi at 1600°F.

Oxidation of columbium and columbium-base alloys

The possibility of developing a high degree of oxidation resistance in columbium by alloying has aroused great interest in the past several years. Columbium oxidizes freely, but forms a very refractory major oxide, Cb_2O_5, which melts at about 1400°C.[16] Correspondingly, MoO_3, the major oxide formed by molybdenum, melts at 795°C. The high melting point of Cb_2O_5 suggests that alloying might produce a more stable oxide film, either by formation of a columbate, a spinel-type oxide, or by other protection mechanisms.

Recent data from three[17,18,19] of the dozen or so research organizations investigating columbium-base alloys confirm that alloying can markedly decrease oxidation at temperatures up to at least 1800°F. Typical oxidation rates found at 1800°F when additions of titanium, molybdenum, vanadium, chromium, and zirconium are added are shown in Fig. 4. The most effective additions create minima which lower the oxidation rate by a factor of approximately 10 at 1800°F, and more at lower temperatures.

In binary alloys with molybdenum and vanadium, catastrophic oxidation occurs when more than the optimum alloying content is added as result of formation of volatile molybdenum and vanadium oxides.

Alloying decreases oxidation of columbium to the level of mild steel. Further improvement, on the order of two magnitudes, is needed before columbium can compare with stainless steel. Thus, the columbium alloys reported in the literature to date would have to be clad for elevated-temperature service if no further improvement can be obtained. However, the low oxidation rate and the absence of a volatile oxide would allow use of much thinner claddings applied with much less care than in the case of molybdenum, where a pinhole can mean failure of the entire clad structure through volatilization of MoO_3.

Columbium, unlike molybdenum, is susceptible to serious contamination-hardening when exposed to oxygen. For instance, oxygen will diffuse rapidly into unalloyed columbium at 1800°F, causing hardness to increase to over 400 Vhn at a depth of about 10 mils in 20 hr. This markedly reduces the useful cross-section of unprotected metal in service. However, several elements that are of interest as oxidation-resistant additions are also quite effective in reducing contamination; these are titanium, vanadium, chromium, and zirconium. Ternary alloying to maximize the best oxidation behavior with the best contamination resistance has not yet been investigated.

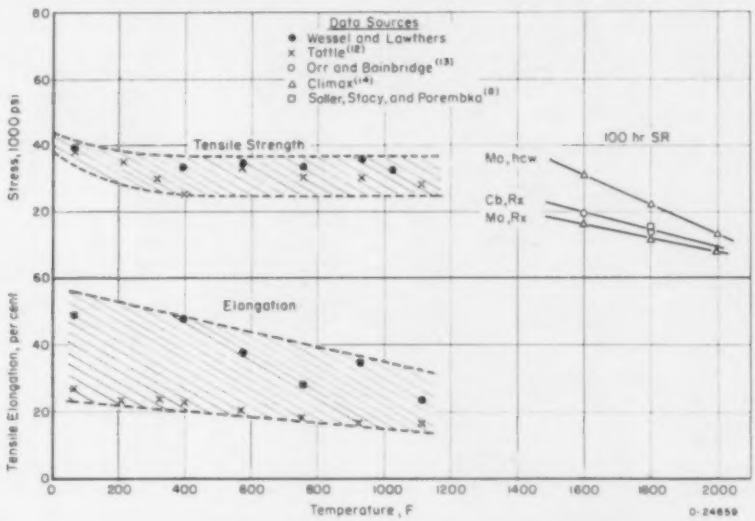

Fig. 3—Elevated temperature properties of columbium. Comparison is made with hot-cold-worked molybdenum, recrystallized columbium, and recrystallized molybdenum.

Table II. Mechanical Behavior of Columbium[8, 11] and Molybdenum Rod

	Columbium	Molybdenum
Room-Temperature Properties		
Hardness, annealed, Vhn	110	187
Hardness, as worked, Vhn	175	255
Ultimate tensile strength, recrystallized, psi x 10^{-3}	39	68
0.2 pct offset YS, psi x 10^{-3}	30	63.5
Elongation, pct	28 (1 in.)	46 (2 in.)
Reduction of area, pct	82	36
Properties at 1800°F		
Ultimate tensile strength, recrystallized, psi x 10^{-3}	17.5 (0.5 hr)*	29.0
0.2 pct offset YS, psi x 10^{-3}	—	10.5
Elongation, pct	47.2 (1.5 in.)*	49.0 (2 in.)
100-hr creep rupture strength, recrystallized, psi x 10^{-3}	14.0*	11.5
Other Properties		
Cold fabricability	Excellent	Fair
Hot fabricability	Excellent	Good
Recrystallization temperature, °F	2200	2300

* Data obtained from strip.

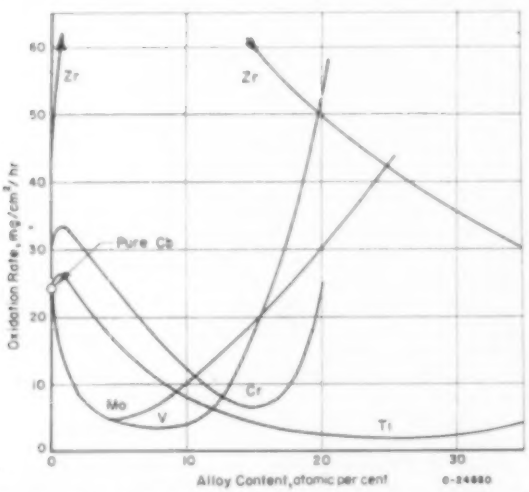

Fig. 4—Effect of alloying elements on the oxidation of columbium in air at 1800°F.[10]

Present uses

A recent appraisal[1] suggests that columbium has probably been distributed as follows among American industry during the period 1955/56:

Ferrocolumbium in Steel (includes ferro-tantalum-columbium)	60 pct
Nonferrous alloys	30 pct
Carbides, pure metal, welding rods, miscellaneous	10 pct

Ferrocolumbium is added to stainless steel (Type 348) to prevent carbide precipitation and chromium depletion. Stainless not stabilized by columbium (or titanium) additions is susceptible to precipitation of chromium carbides in zones affected by welding, which seriously decreases corrosion resistance near the weld. The preferential columbium carbide formation also increases the overall corrosion resistance markedly and improves the strength. Columbium-tantalum stabilized stainless steel (347) is used for the same purpose. Important competitors of the columbium or tantalum stabilized stainless steels are titanium stabilized stainless steel and ELC (extra low carbon) stainless steel. The steels containing columbium are somewhat stronger, more castable, possess a better surface, are superior for nuclear energy applications and have better mill yield. However, users have been slow to return to columbium-stabilized stainless steel following the Korean emergency because of higher price and fear of a recurrence of the late columbium shortage. A few users also prefer the titanium-stabilized stainless steel for technical reasons.

The use of columbium (and tantalum) in nonferrous superalloys as an addition element is popular and has increased in recent years. These alloys, usually cobalt or nickel base, are used for high-temperature applications. Columbium additions are very effective as strengtheners, principally because columbium readily forms stable carbides which increase high-temperature strength and creep resistance.

In addition to these two major uses, relatively small amounts of columbium have been used in cemented carbides for cutting-tool applications, in

Table III. Alloying Behavior of Columbium

Binary Alloying Element	Solidification Behavior: Freezing Characteristic	Solidification Behavior: Lowest Freezing Temp, °F	Known Compounds	Other Features
Aluminum	—	—	$CbAl_3$	—
Carbon	—	—	NbC	—
Cobalt	Eutectic	2260	Co_2Cb, Co_7Cb_2	5 pct Cb solubility limit in α-Co
Chromium	—	—	Cr_2Cb_2	<5 pct Cb solubility limit in Cr
Hydrogen	Eutectic	2550	—	0.04 pct H solubility limit in Cb at 1658°F
Iron	Eutectic	2470	Fe_2Cb_3	2 pct Cb solubility limit in α-Fe at 1650°F
Molybdenum	Isomorphous	4380	None	—
Nickel	Eutectic	2150	NiCb, Ni_3Cb	20 pct Ni solubility limit in Cb at 2320°F
Nitrogen	—	—	CbN	0.03 pct N solubility limit in Cb at 1470°F
Oxygen	Eutectic	2910	CbO, Cb_2O_5	3 pct O solubility limit in Cb at 2910°F
Palladium	—	—	—	σ-phase at 60 pct Cb
Phosphorus	—	—	CbP	—
Platinum	—	—	Cb_3Pt	σ-phase at 62.5 pct Cb
Rhenium	—	—	—	σ-phase at 50 pct Cb
Rhodium	—	—	Cb_3Rh	σ-phase at 60 pct Cb
Silicon	Eutectic	2375	$CbSi_2$, Cb_5Si_3, Cb_5Si_3	—
Tantalum	Isomorphous	4380	—	—
Thorium	Eutectic (β-Th)	2610	—	—
Titanium	Isomorphous (β-Ti)	3130	—	5 pct Cb solubility limit in α-Ti
Tungsten	Isomorphous	4380	—	—
Uranium	Isomorphous (γ-U)	2070	—	Possible eutectoid with β-U
Vanadium	Isomorphous	3290	—	—
Zirconium	Isomorphous (β-Zr)	3160	—	Eutectoid with α-Zr

vacuum tubes, and as an oxygen *getter*. As columbium becomes more well known, incidental uses such as these can be expected to increase.

In the uses discussed above, columbium was not applied as the pure metal except in a few minor instances. But the present wide interest in columbium is being generated by its potential as a pure metal, or as an alloy base, not as an alloying addition.

Future Uses

Atomic Energy: A major use for pure columbium now being exploited is in atomic energy. Its moderately low nuclear cross section combined with potential high-temperature strength make it seem attractive for use close to nuclear fuels in atomic reactors. Thus, it could be used as an ingredient in fuel elements. If the promise of high oxidation resistance can be realized, it could be used in contact with air. Columbium has an excellent resistance to attack by other coolants considered for reactors, such as water, liquid metals, and molten salts. It is compatible as an alloying element with uranium and, does not form brittle compounds when in contact. For these two reasons, it has been chosen by the United Kingdom Atomic Energy Authority as the cladding material for uranium in the Nak-cooled, Dounreay, fast-breeder reactor.

The US Atomic Energy Commission is interested in columbium for similar uses in fast reactors and in thermal-power reactors as an alloying addition. For instance, columbium is an alloying element in the sandwich plate fuel elements of the Argonne experimental boiling water reactor (EBWR).[20] It has been estimated[4] that annual requirements for columbium for domestic atomic power production will range from 5 to 30 tons annually, depending upon the results of present experiments.

Jet Engines: Currently it is believed that cobalt- or nickel-base superalloys (some containing minor columbium additions) can be developed for effective mechanical service in engines for air vehicles at an absolute maximum temperature of 2000°F. Some of these alloys, most of which are quite oxidation resistant, can be used as coatings at slightly higher temperatures when supported by more refractory materials. However, coated materials are limited in applicability, and load-bearing materials, strong, ductile, and oxidation resistant, are badly needed for service at and well above 2000°F.

The highest temperature at which such new materials could conceivably serve is governed only by the properties of the material itself, since design would eagerly be changed to take advantage of higher possible service temperatures. Thousands of tons of metal might be used in this type of service.

Compared with molybdenum and tantalum

Experimental work with molybdenum is being pushed to fit it for service above 2000°F. Its disadvantage of exceedingly poor oxidation resistance and difficulty in fabrication are hard to overcome but, despite this, General Electric Co. recently announced successful testing of a jet engine with molybdenum turbine buckets protected from oxidation by cladding.[21] In many ways, columbium is equally as promising as molybdenum for such applications and, in some ways, apparently superior. This promise has led many commercial organizations to invest in columbium sources, processing, and research.

Columbium, somewhat less dense and having about the same degree of strength as molybdenum at elevated temperatures, can be expected to have about the same strength-to-weight ratio. In addition, as discussed above, columbium is more readily fabricated and considerably more oxidation resistant than pure molybdenum. Unlike molybdenum, alloying increases the oxidation resistance significantly, so that initial work with binary columbium alloys has produced materials with 100 times the oxidation resistance of molybdenum. This is still about 100 times poorer than stainless steels or other significantly oxidation-resistant superalloys. However, much work is being done and significant further improvement can be expected. Columbium may be an answer to the need for a truly self-oxidation-resistant ductile refractory material for ultra-high-temperature service.

Conversely, it should be pointed out that molybdenum is very sharply strengthened by alloying (to the point of unfabricability by present techniques). Columbium is not so responsive. No announcements of alloys with exceptional strength comparable to known molybdenum-base alloys have been presented, but the problem is undoubtedly under intensive study. The high price of columbium is also a major problem, but this situation is familiar. As with molybdenum and titanium, when the metal is studied further new techniques and higher production will cause the price to drop.

Other important new uses for columbium in less dramatic fields are also foreseen. Columbium is nearly as corrosion resistant as tantalum. Since columbium is only about half as dense as tantalum, the same weight will go twice as far in use for some corrosion-resistant applications. As oxidation-resistant alloys are developed, uses such as heater windings, thermocouples, and other similar applications can be considered.

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COMPOUND SEMICONDUCTORS

A survey of the characteristics of compound semiconductors as deduced from measurements of their mechanical, optical, electrical, magnetic, and thermal properties.

by H. P. R. Frederikse

DURING the last ten years impressive advances have been made in the field of semiconductors. Stimulated by technological needs, a considerable amount of research has been concentrated on the elements silicon and germanium. In 1952 several physicists turned their attention to other semiconductors, hoping, that by application of similar approaches, to learn as much about these new materials and to evaluate their possibilities for practical use.

Semiconductors defined

Semiconductors are characterized by their electronic energy band structure, which results from electrons moving through a metal crystal with a *periodic* potential energy. At most electron wavelengths, they are out of phase with each other, but at certain critical values of k, the *wave vector*, they interfere constructively. In terms of the Bragg relation, these values are critical when n=1,2,3, etc. As a consequence, the energy at these levels is either higher or lower than would be expected, giving rise to the energy bands shown in Fig. 1, which is taken from Cottrell.[1]

The width of this gap is one of the most important parameters of a semiconductor and distinguishes it from a metal or an insulator. For metals, the electrons do not fill all the allowed states of the valence band; consequently the gap between the valence states and the conduction states is zero. The difference between semiconductors and insulators is only a matter of degree; the gap in different semiconductors ranges from 0.1 to 4 or 5 ev, a solid with larger energy gaps is called an insulator.

Electrons can be excited across the energy gap by thermal or optical excitation. Once in the higher band, an electron can take part in electrical conduction; the hole left in the valence band contributes also to the current. This process determines the intrinsic conductivity. In many cases, however, the semiconductor contains an appreciable number of impurities (e.g. 10^{16} per cc). These impurities also provide electrons (or holes) for the conduction process. As their activation energy is often very small (e.g. 0.01 ev), the conductivity at low temperatures is usually determined by the impurity concentration.

Another important parameter in semiconductors is the mobility, i.e. the velocity with which the carriers are moving per unit electric field. The mobilities of electrons and holes in germanium and silicon are of the order of several thousand sq cm per volt-sec; in some intermetallic semiconductors the values are even considerably higher.

Classified by position in periodic table and crystal structure

One can classify the compound semiconductors in several groups, depending on the position of the components in the periodic table (shown in Table I) and the crystal structure of the compound. A large number of binaries and ternaries are listed in Table II. The semiconducting elements are included in this table for comparison. The first group is composed of elements from the 3rd and 5th columns of the periodic table and are therefore known as the III-V compounds. Several members of this class have been investigated extensively during the last few years.[2] Some of these compounds (especially GaAs) show potentialities for application as rectifiers and photodiodes; the arsenides and phosphides are, however, more difficult to prepare in a pure state than germanium. InAs and InSb are promising as infrared photodetectors.

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Table I—Extract from the Periodic Table

I	2	3	4	5	6	7	8	0
H								He
Li	Be		B	C	N	O	F	Ne
Na	Mg		Al	Si	P	S	Cl	A
K	Ca						Fe	
Cu	Zn	Ga	Ge	As	Se	Br	TRANSITION	Kr
Rb	Sr						ELEM	
Ag	Cd	In	Sn	Sb	Te	J		X
Cs	Ba						Pt	
Au	Hg	Tl	Pb	Bi	Po	At		

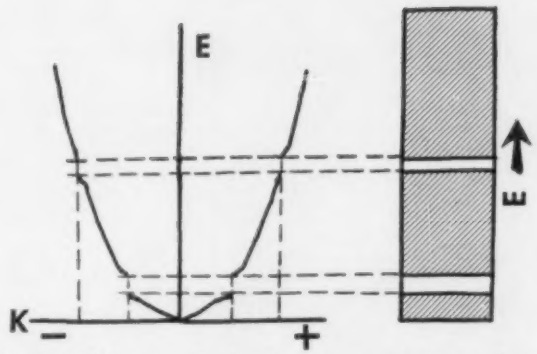

Fig. 1—Left: Energy vs k value for electrons moving in one direction through a crystal; right: Energy bands for this direction (after Cottrell[1]).

The crystal structure of the III-V binaries (zinc blende structure) would be equivalent to the diamond lattice if the two components were identical, Fig. 2. This same lattice structure is also found in nearly all the members of the next two groups: the II-VI and the I-VII compounds. The energy gap of some of these materials is already so large that they are classified as insulators. The best known representatives of this group are CdS and ZnS.[3] The former is extensively used in photoconductive devices, while the latter is a well-known luminescent material.

The next three compounds (II-IV) are examples of crystals with the anti-fluorite structure.[4] This lattice is the inverse of the fluorite lattice (CaF_2) and is very similar to the diamond structure.

The following group, the lead compounds (IV-VI), has drawn much attention, due to their use as infrared photodetector.[5] As evaporated layers, their photosensitivity in the 3 to 6 μ region is larger than that of any other known semiconductor.

The rest of Table II contains some examples of semiconducting materials which have been (re)discovered during the past few years. In most cases the preparation of the compounds is rather difficult, and the information concerning their properties is very incomplete. This is especially true for the ternary compounds listed at the end of the table. The chalcopyrites[6] and the chalcogenides[7] have a crystal structure derived from the diamond lattice, while the I-II-V compounds are related to binaries like Mg_2Sn. It is possible that the importance of these compounds will become greater as knowledge about them increases.

Nature of the chemical bond

One of the most important aspects for an understanding of semiconductors as a whole is the nature of the chemical bond in these solids. Recently several attempts have been made to predict the character of new compounds from the type of binding expected.[8] It has been established that the semiconducting bond is predominantly covalent, while strongly ionic compounds are insulators. We can follow these trends by considering the position of the constituents in the periodic table. The strongly electropositive elements from the first column form heteropolar bonds with the strongly electronegative elements of column 7, e.g. NaCl. In these compounds electron transfer from the column I-atom to the column VII-atom is nearly complete, and the binding force is mainly due to coulomb attraction of the ions. They have large energy gaps and large binding energies.

Going toward the center of the periodic table, the covalent character of the bond increases: the column IV elements are perfectly homopolar. In homopolar crystals the bond is established by an electron pair shared between two neighbors, e.g. in germanium an atom shares its four valence electrons with the four nearest neighbors surrounding it tetrahedrally, see Fig. 2. This type of bonding is—in contrast with the ionic bond—strongly directional. It is also considerably weaker, resulting in smaller energy gaps and smaller binding energies (lower melting points). The covalent bond, furthermore, tends to smooth out fluctuations in the periodic potential of the crystal, also resulting in a decrease of the energy gap.

Similar trends can be observed in the mobility. The thermal scattering of electrons decreases in the sequence α-Sn—InSb—CdTe—AgI, while the polar scattering increases in this series in the same direction. In a compound, like InSb, both polar and thermal scattering are very small, resulting in a very high mobility.

From the above considerations it is clear that the amount of ionic character plays an important role in determining the properties of semiconducting compounds. There are several ways in which information concerning the degree of ionicity, can be obtained, but all of them are approximate.

Departure from stoichiometry

The departure from stoichiometry of semiconducting compounds is an important factor for the preparation of these materials. In some binaries, like InSb and Mg_2Sn, the composition is strictly in accordance with their chemical formula. The phase diagram shows no mixing range, i.e. the solubility of either A or B in the compound AB is negligible. Most compounds, however, (PbS, PbTe, ZnO, TiO_2, etc.) can easily be prepared with an excess of one or the other constituent. The amount of excess can often be controlled by varying the vapor pressure of one of the components. The vacancies (or interstitials) created by such deficiencies can act as acceptors or donors, producing p- or n-type semiconductors.

Electrical conductivity, galvanomagnetic effects

The first measurement usually performed on a semiconductor is a determination of the conductivity and the Hall effect as a function of temperature. From these one can generally determine the energy

Table II. Semiconducting Elements and Compounds

Semiconductor	Energy Gap E_g^0 ev	Mobility μ_n Sq Cm per Volt-Sec	(300°K) μ_p Sq Cm per Volt-Sec	Melting Point, °C	Structure
C (diamond)	6-7	1800	1200		diamond
Si	1.2	1200	500	1414	diamond
Ge	0.78	3900	1900	958	diamond
α-Sn	0.08	3000		260	diamond
Se	2.31		1600	220	hexagonal
Te	0.32	400	1200	452	hexagonal
AlP	3.0				zinc blende
AlAs	2.2			1600	zinc blende
AlSb	1.65	1200	200	1060	zinc blende
GaP	2.4				zinc blende
GaAs	1.45	4000	400	1240	zinc blende
GaSb	0.77	4000	850	702	zinc blende
InP	1.25	3400	650	1070	zinc blende
InAs	0.47	30,000	~200	940	zinc blende
InSb	0.23	65,000	~1000	523	zinc blende
CdS	>2	210		1750	zinc blende
CdSe	1.8			1350	zinc blende
ZnS	3.7			1850	zinc blende
ZnSe				~1000	zinc blende
HgSe	~1.0	>10,000		690	zinc blende
HgTe	~0.15	>10,000		670	zinc blende
AgJ	>2.8	30		557	zinc blende
Mg_2Si	0.77	370	70	1102	anti-fluorite
Mg_2Ge	0.74	520	100	1115	anti-fluorite
Mg_2Sn	0.33	320	260	778	anti-fluorite
PbS	0.37	~600	~250	1110	rocksalt
PbSe	0.25 (300°K)	1200	475	1065	rocksalt
PbTe	0.31	1200	850	904	rocksalt
Mg_3Sb_2	0.82	20	80	1228	complex
Cd_3As_2	0.6				
Cs_3Sb	1.0				hexagonal
Bi_2Te_3	0.2		~250	585	rhombohedral
ZnSb	0.6	60	340	546	rhombic
CdSb	0.52		360	456	rhombic
$CuAlS_2$	~2.5				chalcopyrite
$AgTlTe_2$	~0.1				chalcopyrite
Cu In Se_2 ($I\ III\ VI_2$)	~0.9	300			chalcopyrite
Hg In_2 Se_4 ($II\ III_2\ IV_4$)	0.6				chalcogenide
Li Mg Bi ($I\ II\ V$)	0.4				anti-fluorite

gap E_o and the mobilities μ_n and μ_p of holes and electrons, respectively. A typical example of the results of such measurements is given in Fig. 3 (Mg_2Sn).[11] Most of the values of E_o, μ_n, and μ_p in Table II are deduced from such experiments.

These galvanomagnetic effects also furnish information about a number of other important parameters. The temperature dependence of the mobility depends upon the mechanism of electron (or hole) scattering. At high temperatures, electrons are scattered mainly by acoustical lattice vibrations (in partly ionic compounds the optical branches of the lattice vibrational spectrum play an important role). On lowering the temperature it is found that ionized impurities rapidly take over as the main source of scattering. This occurs usually in the range between 100° and 200°K. At very low temperatures (helium range) the electrons and holes will condense into the impurity states, and neutral impurity scattering will become predominant.

Another important effect is the change of resistivity, $\Delta\rho$, in a magnetic field (magnetoresistance). In first approximation, $\Delta\rho$ is proportional to the square of the mobility. Very large effects are therefore expected for a semiconductor like InSb. While the resistivity change in Ge is only of the order of 1 pct for fields of several thousand gauss, in InSb changes by a factor of 2 are obtained for H = 10,000 gauss at room temperature.[2,12] At low temperatures, $\Delta\rho$ is even larger. By shaping the sample in such a way that one suppresses the Hall emf, the effect can be increased by another factor of 25.[2]

The high mobility of InSb has made possible several applications based upon galvanomagnetic effects.[13] A sample of InSb in an inhomogeneous magnetic field can be used as a transducer of mechanical displacement into changes of electrical conductivity. Other devices making use of the high magnetoresistance are: voltage and current regulators, rectifiers, and amplifiers.

Instead of the magnetoresistance the Hall effect can also be used. The power output from the Hall probes in a given magnetic field is mainly proportional to the square of the mobility. A device based upon this principle is a gauss meter. With proper amplification and field concentration, magnetic fields of the order of 5×10^{-4} gauss are measurable. Another application is the Hall-effect amplifier, Fig. 4. The power output from the Hall electrodes can be considerably higher than the power necessary to produce the magnetic field. Gains of 13 db have been obtained. One of the advantages of such a device is that the power supply is low dc voltage (~0.5 v). The frequency range of operation is, of course, limited by the losses in the magnet.

Rectification and transistor action have been studied in a number of semiconducting compounds[2]. Some 30 years ago several binaries, such as ZnO and PbS (galena), were used as crystal detectors in radio receivers. In recent years some of these materials have been re-studied in the light of our increased understanding of these effects. A number of III-V compounds with energy gaps in the range 0.6 to 1.6 ev show some promise for rectifier application (GaSb, AlSb, GaAs, InP). In particular, GaAs is of interest for use in solar batteries. The energy gap of this material (1.38 ev) is very close to the optimum value (1.50 ev) predicted by theory

for this application. A current-voltage characteristic of a GaAs p-n junction diode is shown in Fig. 5.[9] Such diodes are also sensitive photo elements.

Optical properties

This area of investigation includes optical absorption, photoconductivity, photomagnetic effects, and luminescence. In the limited space available we shall only touch upon a few examples of the first three phenomena.

Optical absorption spectra of a considerable number of semiconducting compounds have now been measured. Most of these materials have their intrinsic absorption edge in the red or infrared part of the spectral range.

A very interesting effect has been found in InSb. The main absorption edge for p-type InSb is always at the same wavelength; for very impure n-type InSb, however, the edge moves toward shorter wavelengths.[14] The explanation of this phenomenon appeared to be rather straightforward.[15] The density of states at the bottom of the conduction band is very small (due to the small effective mass); when the impurity concentration is high, the conduction band will be filled to an appreciable height and optically excited electrons will require a larger energy than that corresponding to the forbidden gap.

Discussion of **photoconductivity** in semiconductors automatically brings the lead compounds in the foreground. PbS, PbSe, and PbTe cells are widely used as photodetectors in the 1 to 7 μ spectral range. The light-sensitive element is an evaporated layer; high sensitivity is obtained by special processes involving oxidation of the layers. Except for PbTe, these cells show good sensitivity at room temperature; they are even more sensitive at low temperatures.

The photoconductive response of the lead compounds shows the usual wavelength dependence: a sharp decrease close to the wavelength corresponding to the energy gap. At room temperature the photoconductive threshold is found at 3 μ (PbS), 4 μ (PbTe), and 5 μ (PbSe). In contrast with most other semiconductors, the energy gap of the lead compounds decreases on cooling. Consequently the limit of sensitivity moves to longer wavelength. The threshold values are at 90°K: 3.8 μ (PbS), 5.1 μ (PbTe), and 7 μ (PbSe).

Work on evaporated layers of the lead compounds predates the study of single crystals by several years. This is due mainly to the fact that the preparation of good crystals is rather difficult. In recent years one has been able to prepare crystals with controlled composition. Their physical properties have been extensively studied. This work has established once and for all the values of the energy gap as listed in Table II.[16]

Photomagnetic effects: The search for far infrared photodetectors has focused attention on several other semiconducting compounds with small energy gaps. In this connection we wish to discuss the photoelectromagnetic (PEM) effect because of its importance for lifetime measurements and its possible application in photoconductive devices.[10,17]

The principle of the PEM-effect is shown in Fig. 6. Infrared radiation, I, is focused on the surface of a semiconducting crystal. As a result, electron-hole pairs will be created which will diffuse into the crystal. A magnetic field, B, perpendicular to the light beam will deflect electrons and holes in oppo-

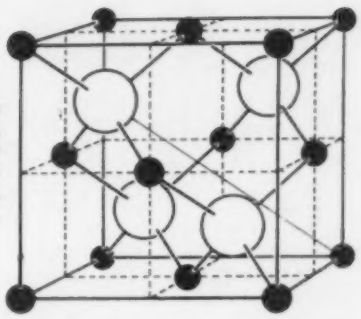

Fig. 2—The zinc blende lattice: solid spheres, Ge, Sb, or Zn; light spheres, Ge, In, or S (after Krebs, *Physica*, 1954, vol. 20, p. 1126).

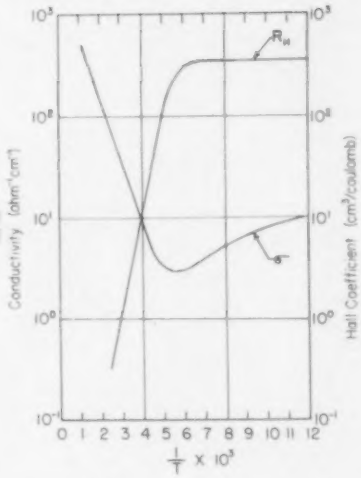

Fig. 3.—Hall coefficient and conductivity of Mg_2Sn.[11]

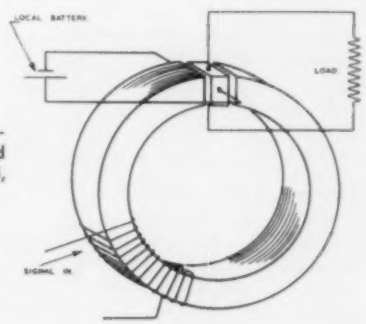

Fig. 4—Hall effect amplifier (after Ross and Shaker, *J. Electro.*, 1955, vol. 1, p. 223).

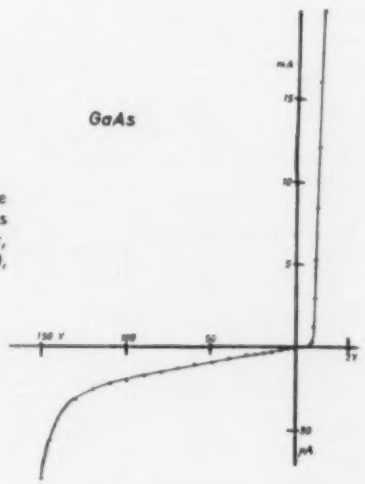

Fig. 5—Current-voltage characteristic of a GaAs rectifier (after Welker, *Physica*, 1955, vol. 20, p. 903).

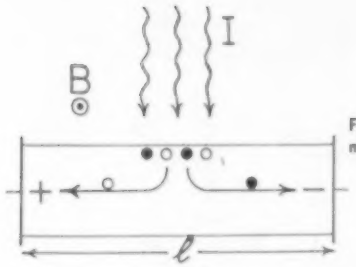

Fig. 6—Photo-electromagnetic (PEM) effect.

site directions. Consequently a (PEM) current will be set up in the a direction perpendicular to both I and B.

This effect has recently been used in the construction of a photodetector for the far infrared. The sensitive element is a thin sample of InSb, and the wavelength limit is 7 μ at room temperature. Although the absolute response is smaller than that of a PbS-cell at 3 μ, its noise-equivalent-power compares favorably with the PbS detector. The sensitivity of the InSb device increases on cooling, but the threshold moves to shorter wavelengths.

The PEM-effect has another important application. It has been shown that combined measurements of PEM-current (or voltage) and photoconductive response provide an easy way of determining lifetimes.[17] One measures the PEM-effect and the photoconductivity of a sample under identical conditions of illumination, first in a magnetic field and then with an electric field bias. The ratio of the two currents yields directly a value of the lifetime τ (providing the mobilities of the carriers are known). This method is especially suitable for lifetimes shorter than 10^{-4} sec.

The scope of this paper does not allow a detailed discussion of lifetimes in semiconductors. We shall quote a few figures to indicate maximum lifetimes observed in certain compounds and elements at room temperature: InSb, 0.1 μ sec; PbS, 40 μ sec; Te, 20-50 μ sec; and Ge, 3000 μ sec.

Thermal properties

Finally, we wish to say a few words about the thermal conductivity and thermoelectric power of semiconducting compounds. In semiconductors, heat is carried almost entirely by the lattice, while the electric current is due to a flow of electrons and/or holes. This is in contrast with metals, where most of the heat is also carried by the electrons. One expects, therefore, that the thermal conductivity of semiconductors will be similar to that of insulators. The few measurements made on compounds confirm these expectations at low temperatures; however, at higher temperatures, several materials seem to have a larger heat conductivity than can be explained on the basis of lattice contributions only. It is rather certain that the higher values are due to an electronic contribution. The theory of this electronic part of the thermal conductivity is, however, not yet fully understood.[18]

Thermoelectric power

The thermoelectric power of semiconductors is several orders of magnitude higher than that of metals. Measurements of the thermoelectric power as a function of temperature are of considerable theoretical interest, especially with regard to the interaction of electrons (or holes) with the lattice vibrations.

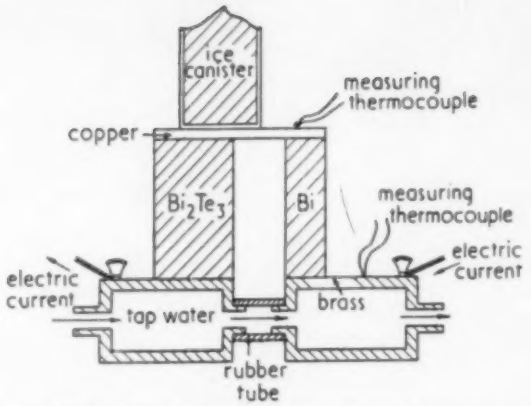

Fig. 7—Thermoelectric refrigerating device using Bi_2Te_3-Bi (after Goldsmith and Douglas (*Brit. J. of Appl. Phys.*, London, 1954, vol. 5, p. 356).

The thermoelectric effect has also drawn attention because of its possible practical use. A thermocouple consisting of a semiconductor and a metal or of two semiconductors usually has a thermoelectric power of several hundred μ-volt per degree. The efficiency of such a couple depends, however, upon the ratio of thermal and electrical conductivities. This ratio is unfortunately, not very favorable for conversion of thermal energy into electrical energy. With the best choice of material (Bi_2Te_3), an efficiency of 6 pct has been obtained for 300°C temperature difference. It is possible, however, that this figure can be increased by using other compounds; it might be that a thermoelectric generator would be feasible as a small-scale power supply.[19]

More promising is the application of the reverse of the thermoelectric effect, the Peltier effect. In this case, a constant current through a closed thermocouple circuit lowers the temperature of one junction with respect to that of the other. Such a thermoelectric refrigerator has been built[20] using Bi_2Te_3 and Bi for the two branches, Fig. 7. This device has produced cooling by as much as 26°C using a primary current of 6 A. Theory predicts that one should be able to obtain a temperature difference of 80°C with suitable materials under optimum conditions.[21]

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DUCTILE CERAMICS — A HIGH TEMPERATURE POSSIBILITY

Condensed from an article by

Earl R. Parker
J. A. Pask
J. Washburn
A. E. Gorum
W. Luhman

Investigations of mechanical properties of some ionic materials has shown the effect of surface conditions on room-temperature ductility.

THE past decade has seen dislocation theory, first conceived in 1934,[1-3] transformed from the state of a hypothetical, imaginative picture into the realm of reality.[2-12] With this maturity there has emerged a clear picture of the physical processes involved in plastic flow, strain hardening, recovery, creep, and solution hardening. Armed with such information, acquired mainly by experimental work on the normally ductile metals, and with some early experimental results which showed that NaCl[13] and AgCl[14] were ductile under certain conditions, it has become possible to predict that ionic solids having cubic crystal structures, normally considered to be brittle, should possess a reasonable degree of ductility at ordinary temperatures.

Joffe's work was starting point

Sodium chloride was first discovered to be ductile by Joffe in 1924.[15] He found that this material, while normally very brittle, became ductile and could be deformed like a metal when immersed in warm water. He attributed this enhanced ductility to the removal of microcracks from the surface of the salt crystals by the solvent action of warm water. Microcracks, he reasoned, would grow easily under stress and, hence, fracture would result before the stresses became high enough to cause plastic flow. Joffe's explanation persisted until very recently, but it was found by the authors and by Dekeyser[16] that Joffe's explanation is not valid. Salt crystals are inherently ductile rather than inherently brittle, as postulated by Joffe. To prove this, one only needs to perform the following simple experiments. Take a single crystal of salt and, with a small sharp chisel and hammer, cleave out a small specimen about $\frac{1}{8} \times \frac{1}{8} \times 1$ in. When all faces of such a specimen are freshly cleaved, the piece can easily be bent by hand. The experiment must be performed quickly, however, because the ductility is an illusive, transient property that disappears in less than a minute after the crystal is exposed to air. Another fact that refutes Joffe's microcrack theory is that the embrittled crystals will actually support a higher load than will the ductile crystals.

The tests reported herein were conducted on the machine shown in Fig. 1 with three point loading; the data are plotted as stress ($S = \frac{Mc}{I}$) vs deflection.

EARL R. PARKER, J. A. PASK, J. WASHBURN, A. E. GORUM, and W. LUHMAN are with the Minerals Research Laboratory, University of California, Berkeley, Calif. This paper was taken from the First Technical Report under contract AF49(638)-56. The complete paper appeared in the *Journal of the American Ceramic Society*, May 1958, p. 161.

All of the face-centered cubic salts tested, except AgCl, show perfect cubic cleavage, so the specimens were therefore made by cleaving. Specimens of materials, such as CaF_2 and CsBr, were shaped with a saw.

Fig. 2 shows typical stress deflection curves for most of the materials tested. These tests were all run in air at room temperature. It is apparent from the curves that all of the materials showed some degree of ductility with the one notable exception, CaF_2. This material exhibited perfect octahedral cleavage and always fractured before bending. It is possible that this material undergoes minute amounts of plastic deformation, but, as yet, it has not been possible to observe any deformation.

Fig. 2 shows clearly that some materials are much more ductile than others. CsBr shows a great deal of ductility; the crystal did not break within the test limits of the machine. Of the materials with the NaCl-type structure, single crystals of LiF are by far the most ductile in air. These results do not in-

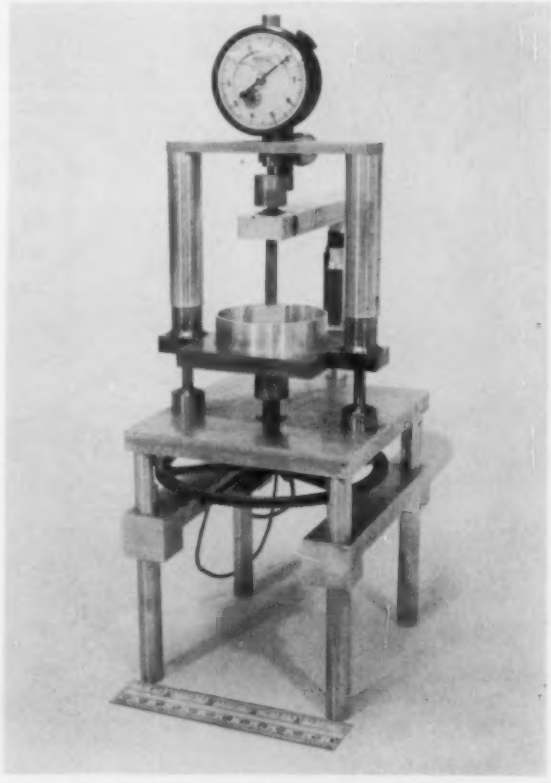

Fig. 1—Bend testing machine for small ionic single crystals.

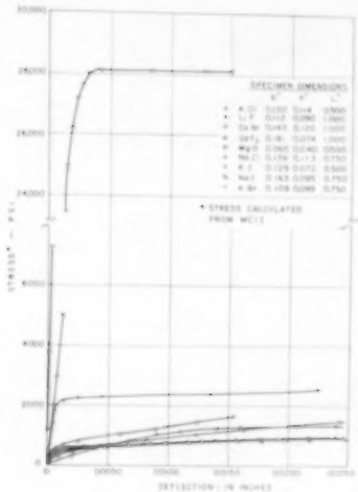

Fig. 2—Stress deflection curves of ionic crystals tested in simple bending in air without previous treatment.

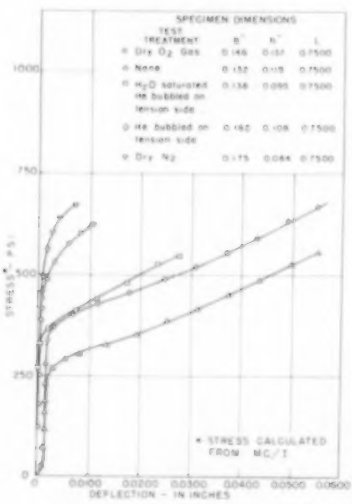

Fig. 3—Stress deflection curves of NaCl crystals cleaved in air and tested under oil.

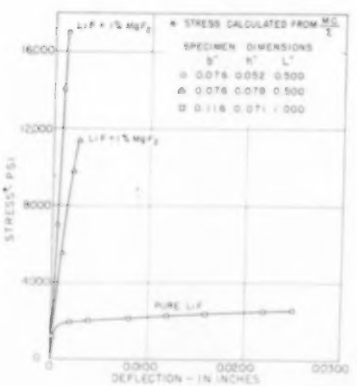

Fig. 4—Effect of 1 pct MgF_2 on LiF.

clude AgCl, which is extremely ductile even in the polycrystalline form. It was found that other materials with this structure would be more ductile if their freshly cleaved surfaces could be protected from air. This was done by immersing the material in out-gassed oils within seconds after cleaving. It was found that the ductility of KCl could be restored by an ammonia treatment.

Air-embrittlement laid to oxygen and nitrogen . . .

The reasons for air embrittlement are not entirely clear, but obviously they have to do with a reaction between the crystal surface and some component of air. Dekeyser attributed the effect to nitrogen,[15] but he did not offer an explanation of how the nitrogen reacted with the salt to cause brittleness. An effort was made to determine the cause of the air effect; thus, specimens were subjected to various atmospheres while being tested. All of the gases, with the exception of oxygen, were deoxidized by passing over red hot iron and then dried in a dry ice acetone trap. Results of the tests on NACl are shown in Fig. 3. Duplicate results were obtained for KCl and KBr. Oxygen and nitrogen are both embrittling agents, but helium and water vapor show very little if any effect.

Only two materials with the NaCl-type structure did not show air embrittlement. These were LiF and MgO; however, MgO seems to become more brittle with time, but the effect is not associated with exposure to air. When tested by hand within one or two seconds after cleaving, MgO is very ductile; however, it soon becomes rather brittle. Test data have not shown this effect because of the long time involved in testing.

. . . and dislocation theory

Just how and why the surface layer affects brittleness is still open to conjecture. It seems likely that this layer is difficult for dislocations to penetrate and so they, unable to escape, pile up at the surface of the reacted layer. Such pile-ups cause the stress in the unslipped portion of the crystal, ahead of the advancing column of dislocations, to increase to approximately n times the average applied stress, where n is the number of dislocations in the array. Experiments have shown that hundreds, or even thousands,[16] of dislocations may function as a unit to cause slip on a single plane. Many of these may accumulate in a single pile up at a grain or sub-boundary;[17] hence, the local stress may approach the theoretical fracture stress and, consequently, a crack may form. There is some evidence that this is indeed the case, but the details are not yet entirely clear. Once a crack has formed, even though microscopic in size, it can propagate in the relatively brittle material even at moderate load levels because of the high stress concentration acting at the root of the crack.

Impurity concentration is a factor

One other major factor controlling ductility is impurity concentration. Fig. 4 shows the effect of 1.0 pct MgF_2 on yield point of LiF. Variation in purity has probably been responsible for the scatter of values for yield of MgO crystals, which are known to contain a minimum of 0.5 pct impurities. The remarkable thing is that the MgO crystals were observed to exhibit significant amounts of ductility in spite of their low purity. It was found possible to bend small pieces cleaved from the crystals through substantial angles without fracture, even at room temperature. Some MgO crystals were brittle and broke with little or no plastic flow. The amount of elongation on the tension side of the specimens varied from 0 to 20 pct, with an average of perhaps 5 pct for the batch tested.

Variations in ductility are to be expected with variations in purity but, as stated previously, it was also found that the ductility decreased with time after cleaving. Experiments to date have failed to disclose the nature of this embrittling action; protection from the air does not forestall the embrittlement. It may be that with this rather impure material, an internal reaction occurs. It is well known[17] that foreign atoms collect on dislocations and that when this occurs, the dislocations are pinned. Dislocation sources may have been activated by the high local stresses that existed during the time that cleavage was occurring, but these sources may have gradually accumulated an atmosphere of foreign atoms and therefore became inactive. Future experiments with purer material will resolve this question.

Preliminary tests have been performed on single crystals of more than a dozen different ionic solids having the NaCl structure. In every case substantial ductility was exhibited, so it can be concluded that ductility is a general characteristic of this class of material. It should not be inferred from these results, however, that ionic solids are as ductile as metals, because they are not. All of them behave in a relatively brittle manner when loaded to failure and they are particularly susceptible to brittle behavior when notched. The metal having properties most closely approximating those of the ductile ceramics is tungsten. This metal is normally very susceptible to brittle failure but, when processed carefully in a certain way, it can be made ductile enough to coil into the tightly wound spirals used for lamp filaments. There is no evident reason why, with proper processing techniques, the cubic ceramic materials could not be made equally ductile in polycrystalline form. In fact it is already known that some polycrystalline materials, e.g., AgCl, can be cast, extruded, rolled, machined, and generally handled like a metal.

As illustrated by Amelinckx[18] and Gilman,[12] materials of this type may be etched to show dislocations. It was found possible to etch MgO with NH_4Cl and H_2SO_4 to give a configuration of etch pits similar to those found in other materials. Fig. 5 shows a typical MgO crystal after bending, cleaving, and etching. A mirror image of the dislocation distribution, as delineated by etch pits, can be seen on the cleaved faces. As with other materials of this type, the slip is on the (110) plane in the [110] direction. If slip were forced to occur on the (100) plane instead of the (110) plane, then like atoms would be forced to approach each other closer, i.e. there would be a higher energy barrier.

Conclusions

Cubic ceramic crystals possess substantial amounts of ductility at room temperature. Even the refractory ceramic material, MgO can be elongated as much as 20 pct on the tension of a single crystal bend-test specimen. Purity and environmental effects play a major role in brittleness. There seems to be little or no reason to doubt that some ductile polycrystalline ceramic materials will be forthcoming within the next few years.

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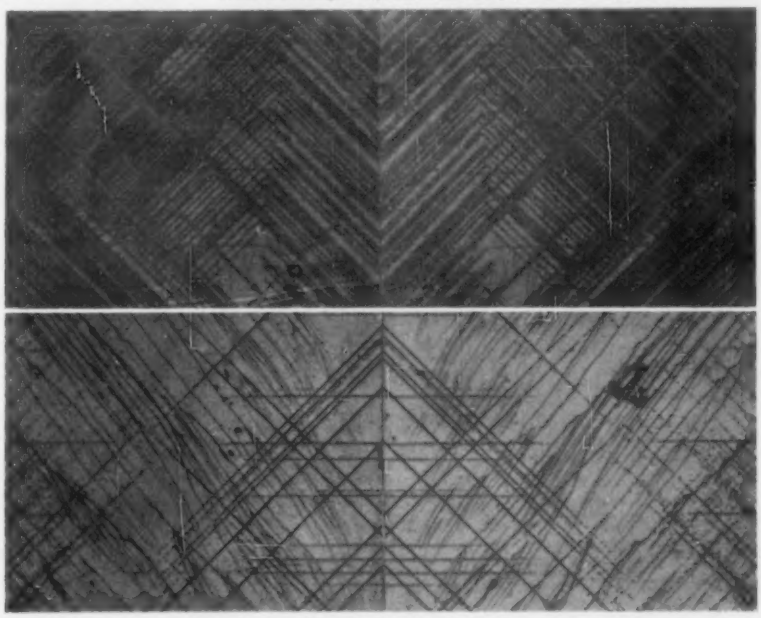

Fig. 5—Single crystal of MgO after bending, cleaving, and etching, showing a mirror image of etch pit configuration. Top: Heavy slipped area near the point of maximum bend. Bottom: In an area of less slip, etch pits are readily observed.

New Directions In Government-Sponsored Research

Last November, Dr. John Chipman, testifying at the Senate Preparedness Subcommittee's enquiry into the missile and satellite program, said, ". . . basic research needs more support than it is getting at the present time . . . the sort of research that has been sponsored in a very laudable manner by the Office of Naval Research, which has been very intelligent in placing fundamental research contracts with the universities." Here, the Head of the Metallurgy Branch of ONR tells which fields are likely to receive emphasis through Government support.

by Julius J. Harwood

THE scientific and technological revolution which this country—and the world at large—are experiencing is, in large part, dependent upon progress in materials research, development, and design. There is no doubt that the limitations of present day materials, and equally important limitations in design and application concepts, represent some of the chief obstacles to the development of new and advanced military weapons, and equipment, and the utilization of nuclear energy, other sources of power, and new industrial processes.

Whether we look at the materials problems associated with jet aircraft and missiles, nuclear reactors, conventional power plants or electronic devices, one common factor is apparent—the need for materials with higher temperature resistance and stability. Breaking through *the thermal barrier* has become an apt expression, indeed.

New demands on materials

The magnitude of the new demands on materials has led to the realization that slight improvements in our present commercial alloys will not be sufficient. Entirely new classes of materials must be made available and radically new concepts in their design and engineering application must be developed. The successful solutions to these complex materials problems will determine, in large part, the rate of advance of our military and industrial technology.

JULIUS J. HARWOOD is Head, Metallurgy Branch, Office of Naval Research, Washington, D.C. These remarks have been abstracted from an address given at the 25th Anniversary of the Metals Research Laboratory, Carnegie Institute of Technology. Opinions expressed are those of the author and do not necessarily reflect the official viewpoint of the Office of Naval Research or the Dept. of the Navy.

The major contribution of basic research in metallurgy lies in providing the solid background of information, both theoretical and experimental, which permit the selection, design, and application of presently available materials with confidence, and which makes possible a rational approach to the search for and development of, new and improved materials.

The science of metals has developed rapidly during the past 15 years—in great part due to the impact of advances in solid state physics. Our knowledge about the structural features of metals and alloys and the factors influencing their mechanical response has reached a high degree of sophistication. There are many, in fact, who believe that we are on the threshold of exciting developments in metallurgy —both from the scientific viewpoint and in the application of new concepts and information for developing alloys of vastly superior properties. However, this tremendous growth in metallurgical science imposes the responsibility of being alert against *diminishing returns* in any given research area or approach. It necessitates a highly critical analysis of research programs in terms of their real potential toward making a significant advance. Acquiring data, per se, is obviously important and often necessary, but of greater importance is the need for bold imaginative research, and the application of new concepts, new techniques, and new approaches. Mobilization of the science of metals to meet the challenge of developing superior materials makes it imperative that metallurgical research not stagnate in the comfort of familiar and over-exploited principles and experimental techniques.

It is possible to extrapolate from certain current trends, and the anticipated needs for materials for military and nuclear power applications, areas of metallurgical effort which are likely to receive future Government emphasis.

High-purity metals

A major effort will be concentrated upon the preparation of high purity metals and single crystals and the study of their fundamental physical properties and metallurgical behavior. The development of more sensitive and reliable analytical methods for impurities and interstitial elements will also be involved. It may well be that a Government supported center (or centers) will be established for this purpose.

High-temperature materials

In the field of high-temperature materials, emphasis will be placed upon:

(a) Exploration of new metal and alloy systems; the refractory metals such as tungsten, tantalum, osmium, iridium, rhenium, etc., to receive particular attention; investigation of methods of preparation, determination of properties, and alloying behavior;

(b) Continued study of the principles of dispersed-phase and fine-particle strengthening of metals, and the application of these principles to practical systems;

(c) The fundamentals of metal-ceramic composite materials, with particular attention to bonding mechanisms and the fundamental factors influencing the mechanical behavior of such composites;

(d) Graphite and, in particular, the problem of surface protection;

(e) Thermal protection systems—the possible use of surface chemical reactions, physical cooling methods, ablation, and combination of materials and techniques to by-pass the fundamental high-temperature limitations of metals and alloys; and

(f) Study of high-temperature thermodynamics and properties of metals and alloys.

Ceramics and inorganic solids

Research on ceramics and inorganic solids will be expanded greatly. Basic scientific information is seriously needed regarding the electrical, thermal, chemical, and mechanical behavior of ceramic-type materials. Particular attention will be placed upon:

(a) The deformation and fracture behavior of simple ceramic compositions;

(b) A systematic study of the structure and properties of high purity intermetallic compounds, as well as oxides, carbides, borides, nitrides, silicides, phosphides, and sulfides, etc.;

(c) Mechanisms and kinetics of phase transformations, precipitation reactions, etc., in ceramic systems, and the relationships between structure, chemical composition, and dielectric, ferroelectric, ferromagnetic or thermoelectric properties of ceramics; such studies to have a particular bearing upon the development of improved high-temperature electronic materials and materials for thermoelectric power devices;

(d) Basic thermodynamic studies involving ceramics, such as gas-solid equilibria in mixed oxide systems; and

(e) Studies of transport phenomena in inorganic solids, i.e., electrical and thermal conductivity, mass transport, or diffusion in oxide systems.

Structure and mechanical properties

Research on the structural features of metals and alloys and the factors influencing their mechanical response will receive additional support, particularly:

(a) Exploration and application of new metallurgical instrumentation and techniques for studying the structure and properties of metals and alloys, e.g., electron microprobe, magnetic resonance, neutron diffraction, isotopic enrichment, etc.;

(b) Elucidation of the fine structure of metals and alloys and the relationships between structures and properties; increased use of *radiation damage* as a tool for studying the imperfection structure of metals and alloys;

(c) Deformation and fracture with particular emphasis on the influence of state of surface and environment on the mechanical response of metals and alloys; increased attention also to be placed on research involving high strain rates and the dynamics of imperfections (dislocations);

(d) High pressure reactions and phase transformations at high pressures and high temperatures;

(e) Transport phenomena in solids, i.e., the mechanism of diffusion, electrical conductivity, thermal conductivity, etc.; and

(f) Exploration of properties and alloying behavior of rare earths and other new metal systems.

Structure and reactivity of solid surfaces

While it has become commonplace to regard corrosion losses as inevitable, the reactivity of materials with high temperature environments, and the corrosion problems associated with propellants, fuels, and nuclear propulsion systems makes this a research area of vital importance, particularly with respect to:

(a) Kinetics of solid-gas interactions at very high temperatures and under high velocity gas conditions;

(b) Fundamentals of liquid metal and fused salt attack on metals and alloys;

(c) Application of new and more sensitive techniques to the study of surface structure and surface reactions;

(d) Effects of nuclear radiation upon the mechanism and kinetics of corrosion reactions and the study of mechanisms of corrosion reactions related to nuclear propulsion; and

(e) Fundamentals of stress-corrosion cracking.

Directions of new research

The above lists represent *some* of the areas of research which are now receiving increased emphasis. Obviously, it is impossible to predict the long-range, future directions of new research—this, itself, is one of the objectives of research. New paths of metallurgical effort will be the result of the contributions of research now underway.

In the more practical vein, it can be stated with certainty that there will be, in the future, a much closer relationship between design and materials. The recognition of temperature limitations of materials and the realization that metallurgical panaceas are not in the near offing, are bringing about long-awaited revisions in materials design concepts. New design and application principles will be developed to take maximum advantage of advances in materials technology.

Discussion

of the article,

Vacuum Melting in the Steel Industry Today

page 1513 of the December Journal of Metals

1) Comments by Professor A. P. Taber of Syracuse University Research Institute

In Fig. 7, subject article, it is noted that the endurance limits for the SAE 4340 air-melted steel, heat treated to 190,000 psi, were 92,000 psi in the longitudinal direction, and only 56,000 psi in the transverse direction, or a ratio of transverse to longitudinal endurance limits of 61 pct. In the tests, at the Syracuse University Research Institute of a commercial SAE 4340 steel, heat treated to various strength levels between 215,000 psi and 269,000 psi, the endurance limits varied between 90,000 psi and 101,000 psi in the longitudinal direction and from 67,000 psi to 74,000 psi in the transverse direction, or the ratio of transverse to longitudinal endurance limits between 70 and 75 pct.

The fatigue tests were performed on a conventional rotary bending machine on specimens machined from 4½ in.-rounds of commercial, electric-furnace heats. The specimens were of the 3 7/16 in. length, conventional type, prescribed in the Metals Handbook. Specimens were rough machined and left approximately 0.015 in. oversize on each surface and were finished to proper dimensions after heat treatment.

The work at this Institute confirms the results of the author that indicate that the vacuum melting of steel results in better mechanical properties. Probably the most important consideration is whether or not for a particular application the improved mechanical properties obtained by vacuum melting techniques offer sufficient advantage to offset the increase in costs over common commercial methods.

In addition to the above mentioned work, Syracuse University Research Institute conducted fatigue tests in bending on notched specimens of SAE 4340 steel and in direct stress on both unnotched and notched specimens of several other commercial type steels, heat-treated to various strength levels between 240,000 psi to 300,000 psi. Table I is a brief summary of the results of the latter tests.

It should be noted that the ratio of the unnotched transverse to longitudinal fatigue strengths in the case of one Tricent and the Super TM-2 are close to that reported by the author for SAE 4340.

Table I. Fatigue Strengths of Conventionally-Melted Alloy Steels

Steel Used	Unnotched Fatigue Strength, Psi		Notched (K = 3) Fatigue Strength, Psi		Ratio Unnotched Transverse to Longitudinal Pct
	Longitudinal	Transverse	Longitudinal	Transverse	
Tricent (Inco)	135,000	80,000	55,000	45,000	59
	120,000	90,000	60,000	50,000	75
	120,000	80,000	50,000	50,000	67
Crucible's SHS-260	90,000	85,000	65,000	60,000	94
	85,000	80,000	40,000	40,000	94
Super TM-2 (Timken)	130,000	80,000	50,000	40,000	62

2) Reply by W. W. Dyrkacz of Allegheny Ludlum Steel Corp.

We wish to thank Professor Taber for the valuable data presented in his discussion. These data re-emphasize the fact that there can be a considerable variation between transverse and longitudinal properties of air-melted materials from heat to heat. This scatter band, to a great extent, is a function of steel melting and solidification variables, such as cleanliness, segregation, cooling rates, ingot size, amount of hot reduction from ingot to bar form, location of test specimens, etc.

The best evaluation of possible property improvements can be made only by comparing consumable electrode remelting versus conventional melting on the *same* heat of material. This was accomplished in the experiment on a heat of SAE 4340 which resulted in the data given in Figs. 7 and 8, p. 1516 JOURNAL OF METALS, December 1957. The heat used was selected at random without any knowledge of the properties beforehand. It is, therefore, gratifying to see that the air-melted quality of this heat is comparable to that of other commercial heats tested at Syracuse University, and that its properties fall on the lower side of the scatter band. Since the greatest improvement in quality is desired in heats of this type, this work demonstrated the extent of property improvement possible in large sections (6 in. sq and 10½ in. sq billets) by consumable electrode vacuum remelting into 16 in. to 20 in. diam ingots of the same air-melted composition. The ratio of unnotched transverse to longitudinal fatigue strength was increased from 61 pct to 81 pct.

It would be most informative to compare ingot size, cleanliness, and reduction from ingot to final bar size of the three commercial steels given in Table I with the mechanical properties shown. Of particular interest would be the ingot size and bar size tested on the SHS-260 steel, since this is the only material which shows a higher ratio of unnotched transverse to longitudinal fatigue strength than the 81 pct obtained with the consumable-electrode, vacuum melted heat of SAE 4340 reported above and in Fig. 7 of the original article.

3) Further remarks by Professor Taber

The Syracuse University Research Institute appreciates the comments of Mr. Dyrkacz on the comparison of properties of the conventionally-melted and vacuum-melted steels. As mentioned previously, we agree, as confirmed by recent work, that the vacuum melting of steel results in better mechanical properties. However, test results are not always consistent because exceptions can be cited where commercially melted steels are found to possess properties superior to some vacuum-melted heats of the same nominal composition.

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AIME NEWS

Society Dinner Features Alaskan Adventures

The Second Annual Dinner of The Metallurgical Society of AIME took place on Tuesday evening, the 18th of February, during the Annual Meeting. The place was the Sheraton-McAlpin Hotel in the heart of New York City. Many of America's leading metallurgists were to be found in the large group which, following dinner, heard the parting remarks of J. C. Kinnear, Jr., who turned over the gavel to incoming President Walter H. Hibbard, Jr.

Two awards were given at the dinner: the **J. E. Johnson, Jr. Award** and the **Robert W. Hunt Award.** Recipient of the Johnson award was Richard H. White, Supt. of Blast Furnaces, Fairless Works, U. S. Steel Corp. The citation prepared for Mr. White reads, "For his contribution in obtaining record production rates on blast furnaces and for his substantial contribution to a practical process of desulfurizing hot metal which could, in the future, permit even larger production rates."

The Hunt Award is given annually for the best paper on iron and steel. This year's Hunt medalists were George W. Healy and Donald C. Hilty, both on the research staff of the Electro Metallurgical Co., Div. of Union Carbide Corp. The paper for which the award was given was titled, *Effect of Oxygen Input Rates in the Decarburization of Chromium Steel* published in the JOURNAL OF METALS in May 1957.

Entertainment highlight of the evening was the talk of Harold Eide, who recounted the adventures of a lifetime spent in the search of gold in Alaska, fortunes won and lost, and tales of what was America's last frontier. Mr. Eide still returns to Alaska during the summer, but now spends the winter on the lecture route rather than the Arctic Circle, and judging by the warm reception given him at the conclusion of his talk, his winters are well spent.

Dr. Gerhard Derge Appointed Editor of New Transactions Bimonthly

G. DERGE

Dr. Gerhard Derge, Jones and Laughlin Professor of Metallurgy at the Carnegie Institute of Technology, has been appointed Editor of the TRANSACTIONS OF THE METALLURGICAL SOCIETY OF AIME, it has been announced by Walter R. Hibbard, Jr., President of The Metallurgical Society of AIME.

In his new position, Dr. Derge will supervise and coordinate the review and selection of manuscripts that are to appear in the new bimonthly journal. In this capacity, Dr. Derge will be working closely with the three Divisional Publications Committees representing iron and steel, extractive metallurgy, and physical metallurgy. Dr. Derge will handle the new job from his Carnegie Tech office, where he will continue his other duties.

The appointment represents another step in the fulfillment of the Publications Policy Report, endorsed by the Board of Directors last November. That report called for an editor who was a professional metallurgist of undoubted reputation to fill the newly created post. John Chipman, Morris Cohen, and J. Herbert Hollomon were appointed as a nominating committee to find the right man for this job. In selecting Dr. Derge, they have found a man who satisfies every qualification that the report put forth.

Born in Lincoln, Nebraska, in 1909, Dr. Derge received his education at Amherst and Princeton, holding a research fellowship at the latter institution, where he received his PhD in 1934. He holds a Phi Beta Kappa key and also belongs to Sigma Xi.

From Princeton he went to the Metals Research Laboratory at Carnegie Tech, where he has been ever since. Holding a succession of higher positions, he was, in 1951, the first person appointed to the newly created Jones and Laughlin professorship, which he still holds.

He was elected to the Institute in 1934, and his application for membership bears the signatures of R. F. Mehl, C. S. Barrett, and F. N. Rhines. Dr. Derge also holds membership in the American Chemical Society, American Society of Metals, and the Electrochemical Society.

Dr. Derge needs no introduction to members of the Iron and Steel Division, where he has been active on technical and publications committees for many years. He has also served as a member of the former Technical Publications Committee. He has written over seventy technical papers which have been published in various journals. In 1952, he was the recipient of the Robert W. Hunt Award for the best original paper on iron and steel. His special field of interest has been in the physical chemistry of blast furnace, open hearth, and bessemer steelmaking operations.

ANNUAL MEETING DEFIES BLIZZARD

Arrival by dog sled was almost the order of the day as the 1958 AIME Annual Meeting got underway in New York in the midst of the season's heaviest snowstorm which had succeeded in grounding planes, delaying trains, and generally making travel an awesome experience for all except the heartiest AIME members from northern ice floes.

Registration opened on Sunday afternoon the 16th of February—The Metallurgical Society of AIME at the Sheraton-McAlpin Hotel and the Society of Mining Engineers of AIME and the Society of Petroleum Engineers of AIME at the Statler Hotel.

The Convention Manager—there himself because he had remained over night at the convention hotel—and a few hardy souls manned the registration desk. Having been used to smashing all previous attendance records time upon time, it was a new experience for him to rush up to the first bewildered registrant—shaking snow from his fur cap—and grasp him with both hands in warm welcome. Soon another icy face was seen approaching, and along with the first snow plow familiar faces began to appear one by one.

Monday, the 17th of February, saw a few frantic phone calls received from authors stranded in far flung villages, but the volume of registrants began to grow, and by Tuesday things began to look like what we have been accustomed to see as the vast throng of metallurgists, mining engineers, and petroleum engineers gather once a year under the AIME banner. Final attendance of 3080—excluding the ladies—may not have been a record, but those who braved the elements to get there, found the Annual Meeting well worth their effort.

Technical sessions, which produced papers giving details of smashing metallurgical developments, were well attended and included many lively discussions. Business meetings produced fruitful developments along the growth path of The Metallurgical Society of AIME. Social events—in contrast to the zero temperatures outside—produced a warm and gay atmosphere for seeing old friends and making personal contact with colleagues of the metallurgical profession. It is to the credit of the New York section and the convention staff that, in spite of all handicaps of weather, that the marathon AIME meeting went on at two of the city's leading hotels without a hitch.

Activities actually began on Saturday afternoon with a meeting of the 1957 Board of Directors of The Metallurgical Society to finish up the year's business. Sunday's events featured a Membership Committee Meeting in the morning and a symposium on Metallurgical Manpower in the afternoon.

Welcoming Luncheon Hears of Record-Breaking Membership

The Ballroom of the Statler Hotel in New York City was the scene of the Welcoming Luncheon of the 1958 Annual Meeting. Tables were well filled with an overflow into the balcony. AIME President, Grover J. Holt presided, announcing that 1957 had netted an increase of 3000 AIME members, thus making 32,800 as of the first of January. Honored guest, Horace J. Fraser, president of the Canadian Institute of Mining and Metallurgy, brought greetings to all AIME members from colleagues across our northern border.

Prizes were awarded in the Institute's Sixteenth National Student Prize Contest, the details of which had been supervised by the Subcommittee on Student Prize Awards with John Butler as Chairman and A. P. Woods for metallurgical papers. The awards are $100 cash prizes for papers in two classifications: those by graduate student members and those by undergraduate student members. For The Metallurgical Society of AIME the winner in the undergraduate division was William J. Babyak for his paper, *The Relation Among the Grain Boundary Area, Twin Boundary Area, and Hardness of Recrystallized Cartridge Brass*, an abstract of which was published on p. 199 of the March issue of JOURNAL OF METALS. The paper was entered by the Pittsburgh section.

Each year the Institute conducts a membership application contest among local sections. The winners are chosen on the basis of the greatest number of applications submitted in proportion to the section's membership. This year's recipients, which were presented the President's Banner at the Welcoming Luncheon, were the following sections: Upper Mississippi Valley, Western Venezuela, Denver-Petroleum, Permian Basin, and Dallas. Membership chairmen should take note . . . not one of these sections has a membership that is predominantly metallurgical!

William J. Babyak, (right), was congratulated by Grover J. Holt, (left), AIME President, upon winning the Student Prize Paper Award, presented at the Welcoming Luncheon, Monday, Feb. 17, 1958.

For the submission of the largest number of applications for Student Associate membership, the Chapter of the Texas Technological College received a prize of $100.

Speaker at the Welcoming Luncheon was Detlev W. Bronk, President of the National Academy of Sciences and National Research Council and President of the Rockefeller Institute for Medical Research. Dr. Bronk opened his remarks by telling of his overnight trek to New York from his home in Pennsylvania, starting out on snow shoes, followed by a lift on a snow plow, and only later getting to the more conventional modes of transport. Using this example of dependence upon transport media, Dr. Bronk pointed out the essential personal nature of scientific development to us all. He emphasized that we had been neglectful in not allowing science to be an intimate and respected part of our life and culture, and concluded by warning that we must do everything possible to keep alive the creative desire, if we wish to keep our Nation young and strong.

Listening, while Detlev W. Bronk, (standing center) delivered the Welcoming Luncheon Speech, were left to right: Horace Fraser, President of the Canadian Institute of Mining & Metallurgy; and Andrew Fletcher, Past President of AIME.

Top, left: Richard H. White, the recipient of the *J. E. Johnson, Jr., Award*, presented at The Metallurgical Society Annual Dinner on Tuesday, Feb. 18, 1958.

Bottom, left: Harold B. Emerick, Chairman of the Institute of Metals Division, presents the *Robert W. Hunt Medal* to D. C. Hilty, and G. W. Healy, at the Annual Dinner on Tuesday.

Top, right: Shown speaking during the ISD Luncheon, Feb. 18, 1958, is K. L. Fetters, the incoming ISD Chairman. Seated at the guest table were, left to right: Tom C. Campbell, Editor-in-Chief, The Iron Age; Dr. Maxwell Gensamer, Howe Memorial Lecturer; and Mrs. Gensamer.

Metallurgical Manpower Symposium

Although both of the associate chairmen were snowbound, the Metallurgical Manpower Symposium got underway as scheduled, under the capable direction of R. Schuhman, Chairman of The Metallurgical Society's Education Committee.

In the keynote address, H. A. Meyerhoff, a member of the Scientific Manpower Commission, discussed recommendations that the Commission was making in regard to what it felt should be the Government's role in aid to education.

The major part of the program was devoted to the report of the Committee on Manpower for the Metallurgy and the Ceramics Professions. The problem under discussion was what these professions, themselves, are going to do toward increasing the interest of young people in their fields.

The problem was divided into both adult and student awareness, these facets being discussed by W. R. Hibbard, Jr., and W. E. Mahin, who served as Secretary of the Metallurgy and Ceramics Manpower Committee.

The program for action, as presented by G. A. Roberts, former President of the ASM, revolves around establishment of the Metallurgy-Ceramics Foundation, a nonprofit organization devoted to bringing the various recommendations of the Committee into reality. Plans for the Foundation are still in the formative stages, and will be reported later in the year.

Council of Education, Successful Session

Considering the weather conditions, it was most surprising to see the Georgian Room of the Statler Hotel filled to capacity for the Council of Education's Social Hour, Buffet Dinner, and Evening Session. Educators from as far away as California were in attendance, and the social hour provided a welcome opportunity to renew old friendships.

The session after the dinner was under the joint chairmanship of J. C. Calhoun, Jr., and Koehler Stout; the featured speaker of the evening was Roger V. Pierce, Consulting Engineer from Salt Lake City, who reviewed the educational problems facing the profession in a talk entitled, *The Mineral Engineer and What's Next.*

Stag Dinner-Smoker

On Monday evening the social program of the 1958 Annual Meeting got underway with glasses lifted in the Pre-Smoker Cocktail Party, held atop the Statler Hotel, overlooking Pennsylvania Station. The cocktail party was free to all registrants thanks to the sponsorship of a number of companies.

Properly warmed up, registrants moved in the direction of the Ballroom of the hotel where a splendid dinner and stag show awaited them. Varied was the word for the entertainment that followed the turkey dinner. First to greet the *aye* of the well wined and dined throng was a charming mistress of ceremonies who was fitted out in a costume that was in itself quite a piece of engineering. This received the enthusiastic approval of the crowd, as did the succeeding acts which featured a variety of song, dance, acrobatics, and comedy.

Howe Memorial Lecture

Highlight of Tuesday morning's sessions for The Metallurgical Society of AIME was the Howe Memorial Lecture, this year given by Dr. Maxwell Gensamer. Title of the lecture was *The Structure of Metals and the Strength of Structures.* It will appear in the Transactions of The Metallurgical Society of AIME and in excerpted form in the JOURNAL OF METALS later in the year.

Howe Memorial Lecturer, Dr. Gensamer, received a B.S. and a M.S. in Metallurgical Engineering in 1924 and 1932, respectively, from Carnegie Institute of Technology. In 1933 he received a Doctor of Science degree. With a background in industry and in teaching, he is currently Professor of Metallurgy in the School of Mines at Columbia University, where he also serves as Associate Dean of the School of Engineering. He is author of the book, *Strength of Metals under Combined Stresses.*

ISD Luncheon

The first annual ISD Luncheon and Business Meeting, held on Tuesday the 18th of February, was presided over by 1957 ISD Chairman, H. B. Emerick. He announced that 2258 members of The Metallurgical Society of AIME had chosen the Iron and Steel Division as their major technical interest area, an increase of 110 over the previous year. Mr. Emerick reviewed the 1957 regional and national meetings of the division as well as significant publications sponsored by ISD during the preceding year. K.L.Fetters, formally installed as 1958 ISD Chairman, spoke briefly. The main speaker was Tom C. Campbell, Editor in Chief of The Iron Age; his subject was, *What's Ahead for the Steel Industry in 1958-59.* He predicted a modest rise in steel production by mid April with a considerable increase in buying by May or June in anticipation of a price increase by the steel industry on July 1st.

A. H. Cottrell Delivers IMD Lecture

Dr. A. H. Cottrell of the Atomic Energy Research Establishment, Harwell, England, delivered the annual Institute of Metals Division lecture. Entitled, *Theory of Brittle Fracture in Steel and Similar Metals,* the complete text of Dr. Cottrell's paper appeared on p. 192 of the April issue of the TRANSACTIONS OF THE METALLURGICAL SOCIETY OF AIME.

After the Annual Meeting, Dr. Cottrell made a cross-country tour of metallurgical laboratories, including Bell Telephone, California Institute of Technology, the University of California, the University of Illinois, General Electric Research Labs, Harvard University, and the Massachusetts Institute of Technology.

All Institute Session

Tuesday afternoon was reserved for the second annual All-Institute Session. Colonel C. G. Patterson, Chief, Department of the Army Headquarters, Army Ballistics Missile Agency, Redstone Arsenal, Alabama, spoke on *Push Buttons in Modern Warfare.*

Col. Patterson told AIME members that "The radio data signal being received from Explorer I serve notice that the first bold step has been taken by us in the conquest of outer space. But, in the meantime, the Sputniks and the Mutniks have served notice that perhaps we have been guilty of an expensive and time-honored luxury of underestimating our adversary and believing we always would have the first, best, and most of everything.

"Perhaps we have hypnotized ourselves into believing that a nation which was bypassed by the industrial revolution and knows nothing of the rights of the individual as we enjoy them could have no technology and, therefore, was incapable of competing with our vast industrial machine. Have we forgotten that some of the most dynamic new ideas which have influenced mankind have originated in societies which knew no individual freedom such as we have in the Western World.

"Much has been and much more will be learned from the data radioed back from Explorer I. With the information thus gathered we will have a better basis on which to engineer future satellites—and rest assured there will be more and larger US earth satellites in the weeks, months, and years ahead The conquest of space offers a real challenge, one which we can and do accept."

At 4:00 p.m. on Tuesday the 18th of February the Annual Business Meeting of AIME was officially called to order by President Grover J. Holt. There were reports of the Secretary of AIME, the Treasurer, the Business Manager, the Institute Activities Manager, the Chairmen of the Admissions Committee and the Chairmen of the Membership Committee, the secretaries of the three societies, the Editorial Director, and the Field Secretary.

EMD Luncheon

The Annual EMD Luncheon and Business meeting was held on Wednesday the 19th of February with Bruce W. Gonser, Division Chairman, presiding. J. C. Kinnear, Jr., past chairman of EMD and 1957 president of The Metallurgical Society of AIME, was presented with an engraved tin plaque in commemoration of his service to EMD.

The EMD Award is given for the best paper in the field of extractive metallurgy. Chairman of the EMD Award Committee, John D. Sullivan, presented the gold medals and the certificates to John N. Ong, Jr., Milton E. Wadsworth, and W. Martin Fassell, Jr. for their paper, *Kinetic Study of the Oxidation of Sphalerite,* which was published in JOURNAL OF METALS, Feb. 1956.

All three recipients received their Ph.D. degrees from the University of Utah: Fassell in 1949, Wadsworth in 1951, and Ong in 1955. W. Martin Fassell, Jr. later became Associate Professor at the University, and in 1955 he joined the Howe Sound Co. as director of research. Milton E. Wadsworth has remained at the University where he is now Associate Professor. John Ong, Jr. worked for a time with the Uranium Corp. of America, later becoming Assistant Professor of Mechanical Engineering at Washington University, St. Louis.

Dean C. L. Wilson of the University of Missouri was principal speaker at the EMD Luncheon, his topic, *Short Cuts.*

In the business session the chairman commented on the reorganization of some of the technical committees and the adding of new ones to secure adequate coverage of the extractive metallurgy field. T. D. Jones was selected as treasurer of EMD. The new chairman of the Division, Herbert Kellogg, professor of metallurgy at Columbia University, was introduced and closed the meeting.

Annual Banquet Held at Waldorf

More than 2500 gathered in the Grand Ballroom of the Waldorf Astoria Hotel for this year's Annual Banquet. Scores of the nation's top engineers, industrialists, educators, economists, scientists, and public officials were in attendance as the highest awards presented by the Institute were given out by retiring AIME President Grover J. Holt.

Honorary Membership, the highest distinction in the Institute, was awarded to Donald Hamilton McLaughlin, Champion Herbert Mathewson, and Fred Searls, Jr. Their biographies appeared in the February JOURNAL OF METALS.

This year's Hoover Medalist was Scott Turner, former Director of the US Bureau of Mines and a former AIME President. His citation reads, in part, "Distinguished Mining Engineer of international experience and reputation, . . . active in promoting sound engineering practices and ethics and the sense of public responsibility."

Fred D. De Vaney, Director of Metallurgy and Research with the Pickands Mather & Co., was honored with the Richards award for his developmental work in methods for taconite beneficiation. Mr. De Vaney's latest paper, on pelletizing in shaft furnaces, appeared in the February JOURNAL OF METALS.

Other recipients of awards at the Annual Banquet have been reported in the January, February, and March issues.

Concluding the program, Grover J. Holt turned the President's gavel over to incoming President Augustus B. Kinzel, who made a brief address and adjourned the meeting.

The banquet was followed by a reception and dance.

Souvenirs of the banquet, Linde star sapphires, were contributed by the Union Carbide Corp.

Institute of Metals Division Session Reports

High Speed Stressing of Solids—In spite of the unfortunate absence of Professor Davies, due to sickness, and Professor Wood, due to the weather, an interesting and informative session was held on Monday morning.

Professor Cyril S. Smith of The University of Chicago agreed at very short notice to give a lecture during the morning session. His lecture dealt with the characteristics of shock waves in steel and copper, the effects of shock waves on the structure, and the possible mechanisms by which these effects occur.

John Rinehart of the Smithsonian Astrophysical Observatory at Harvard University, Cambridge, spoke on the *Fracture of Metals* caused by explosive loading. Dr. Rinehart discussed the stress conditions arising from the super-position of compression shock waves and the tension waves produced by reflection. He showed numerous examples of the types of fracture that are produced under these conditions and explained how they could be accounted for.—*Bruce Chalmers.*

Diffusion and Imperfections—The session on Monday morning was short several papers and a chairman, presumably because of the weather. Nevertheless, average attendance was close to 100. Under the chairmanship of R. W. Balluffi and P. Shewmon the remaining papers and discussions enjoyed the benefits of extra time.

The papers as a group dealt primarily with the behavior of vacancies in metals and alloys—their mobilities when in thermal equilibrium, their retention on quenching, and their elimination on annealing. The interactions between vacancies and dislocations, especially the formation or migration of dislocations, were considered. However, the most active discussion followed the paper of Lee and Maddin in which an increase in vacancy concentration was inferred

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Shown above, left, Dr. John D. Sullivan, Battelle Memorial Institute, presents the Extractive Metallurgy Division Award to John N. Ong, Jr., Washington University. The other recipients of this Award were, left to right: Milton E. Wadsworth, University of Utah; and W. Martin Fassell, Jr., Howe Sound Co. Below, left, EMD Chairman, Bruce W. Gonser, presents a plaque to J. C. Kinnear, Jr., past chairman of EMD, and 1957 President of The Metallurgical Society of AIME, in commemoration of his service to EMD.

IMD Session Reports

(Continued from page 362)

from the observation that the self-diffusion coefficient in silver increased linearly with the rate of twisting a silver wire. The activation energy was observed to decrease with increasing strain rate; it was about 44 kcal per g-atom at zero strain rate and leveled off at about 17 kcal per g-atom at a strain rate of 20×10^{-5} per second.—*C. B. Birchenall.*

Impulsive Loading—This session of contributed papers on Monday afternoon was also well attended, as many as 160 persons being in the audience. The speakers described means of producing very rapid rates of loading, including the use of bullets fired at the samples and explosive charges set off in contact with the samples. In many instances shock-waves were produced in the metals, and their velocity of propagation was observed. These extreme methods of testing were shown to be remarkably useful in studying fracture, delay-times in yielding, dislocation motion, and other material properties.—*B. Chalmers.*

Deformation—This session on Monday afternoon was attended by as many as 125 people in spite of the severe storm.

A discussion of deformation does not take place in these days without the presentation of new knowledge about dislocations; this session was no exception. Hans Conrad presented two papers, one of which was with W. D. Robertson. From creep and tensile results over a wide range of temperature he analyzed the process of deformation and showed there was some agreement with a rate controlling mechanism based on the formation of thermal kinks in dislocations lying in close-packed directions. The typical wavy slip lines of iron were explained by J. R. Low, Jr. from studies of the dislocation structure of slip bands to be due to screw components of dislocations following curved paths. J. J. Gilman in continuing his work with LiF crystals showed that screw dislocations produce cleavage steps on fractured surfaces, and he presented further evidence for the phenomenon of crack nucleation of dislocations; Norman Brown chalked out an analysis to show that the macroscopic yield point in zinc is associated with the resistance of the lattice to dislocation movement.

Despite the popularity of dislocation research, critical resolved shear stress and strain-hardening behavior were not forgotten. U. F. Kocks discussed work, with R. S. Davis, on single and bicrystals of aluminum to show the orientation conditions under which bicrystals harden more than single crystals. Critical resolved shear stresses were reported to be comparable in shear and tension tests on aluminum by W. L. Phillips, Jr. from a study with W. D. Robertson. Interesting facts were presented on the effect of abruptly changing the slip direction in an interrupted test on a single crystal. H. G. F. Wilsdorf presented new information obtained with J. T. Fourie about slip lines in α-brass from electron micrographs. E. J. Rapperport reported on preparing crystals of zirconium and establishing the room temperature slip and twinning systems.—*W. C. Ellis.*

Mechanical Properties—About 100 people heard six papers covering ferrous, non-ferrous and rare metals, at this session on Tuesday morning.

F. M. Yans described how copper increases the strength with no apparent decrease in reduction in area of oriented beryllium sheet. No explanation for this effect was proposed; the effect of decreasing oxygen content was not investigated.

A. J. Griest described how the uniform elongation of alpha-beta titanium alloys is dependent upon both solution treatment temperature and alloy content of the beta existing at the solution temperature. Close control of solution treatment temperature is required to obtain maximum formability of these alloys.

J. L. Rutherford showed that the higher the purity of zone-refined iodide titanium, the better the low temperature ductility and the lower the flow stress. Greater ductility at −196°C than at −269°C was observed; which is unexplained.

J. C. Wijman showed that there is a discontinuity in the true stress-true strain curves of polycrystalline iron which coincides with the general onset of cross-slip. The contoured type specimen used in this study is a powerful research tool for studies on iron and will undoubtedly see much further use.

A. P. Coldren described how the creep rupture life of some heat-resisting alloys is strongly dependent upon percent reduction during hot rolling whereas others are more dependent upon temperatures of rolling than amount of reduction.

A. H. Daane described a method for preparation of Europium metal and gave data on several of its physical properties. It was concluded Europium behaves as expected of a rare earth metal.—*J. M. Roberts.*

Structural Properties of Metals and Alloys—About 100 persons attended this Tuesday session. Discussion was principally concerned with three papers 1) *Effect of Orientation on Strain Induced Boundary Migration in Silicon-Iron Bicrystals*, 2) *Solubility and Precipitation of Vanadium Nitride in Alpha and Gamma Iron*, and 3) *Mechanism of Age Hardening in Magnesium-Zinc Alloys.* In the discussion to the first paper, a question was raised as to the influence of substructure adjacent to the grain boundary on boundary movement.

The influence of vanadium on reducing solubility of nitrogen in steel (paper two) stimulated a good deal of comment since there is considerable significance to steel technology on the results. Questions were raised as to the relation of thermodynamic properties to particle size of VN and as to whether the results could throw any light on the fact that with increasing purity of iron there was diminished solubility of oxygen.

In regard to paper three, the discussion was concerned with the interpretation of electron diffraction data. The points raised were concerned with the actual identity of the precipitate, as MgZn ore magnesium-modified $MgZn_2$ structure. The interpretation of the hexagonal symmetry of diffraction spots as indicating rods or plates was raised. The author suggested that rods were oriented normal to the base plane, whereas the alternate possibility of plates parallel to the plane was brought up.—*H. Margolin.*

General Metallurgy—This Session covered a variety of topics ranging from the hydrogen embrittlement of titanium to the creep of zinc single crystals.

Lammel and Chalmers discussed their studies on mass transfer between liquid and solid metals, which involved some clever experimental techniques. In a static system, as would be anticipated, the reaction is diffusion controlled, whereas in the case of stirring, the data indicated a surface reaction controlling step. An orientation effect was also observed, in that fine grain material went into solution twice as fast as coarse grain samples. Solid lead-liquid tin was employed as the experimental system. Studies on the solid solubility of nickel in beryllium, reported by S. H. Gelles, lead to the suspicion that there may be an intermediate, Be-Ni phase, which was previously unreported.

A fairly detailed study of the Ni-Ti-C system was presented by E. R. Stover. One of the more interesting items is the fact that within a certain range of nickel composition, and temperature, graphite can be precipitated from solution. Betterton and his associates at Oak Ridge National Laboratory have undertaken a broad fundamental alloying program utilizing zirconium alloys as the base metal system. Ag-Zr, In-Zr and the Sb-Zr systems have been studied to date. The importance of impurities in causing marked deviation from the equilibrium phase limits was one of the more obvious conclusions evident in this work. Zirconium may be considered to be divalent in the solid state; lower valence alloying elements, e.g. Ag, lowers the phase transition temperatures — whereas

higher valence elements e.g. Sb, raise the transition temperature. Atomic size and electron concentration also are significant factors in such considerations.

Dr. H. Steuwe of the University of Illinois, presented some results on the flow behavior of zinc single crystals which appear to confirm Cottrell's theory of dislocation locking by solute atmospheres.—*J. J. Harwood.*

High Temperature Alloys—A great interest in the effect of processing variables on the properties of high temperature alloys was shown by the approximately 200 people attending the session. In the opening talk, F. S. Badger provided an historical review of the development of high temperature alloys. It was emphasized that new developmental approaches would be necessary to achieve the substantially higher operating temperatures required by designers and outlined the importance of work on improved melting methods.

The importance of this statement was attested to in the next three papers by E. E. Fletcher and A. R. Elsea, by J. A. Miller, and by P. E. Hamilton, E. L. Bolin, and R. E. Roth. Small concentrations of gaseous impurities greatly affect properties. It was inevitable that a lively discussion would be carried on concerning the effect of vacuum melting and pouring.

Much interest was shown in the possibilities of the slip casting method as described by L. M. Schifferli, Jr. for the production of articles of refractory materials. Apparently some of the discussors considered it a possible answer to production problems.

Boron and zirconium addition to high temperature alloys were shown by R. F. Decker and J. W. Freeman to prevent intergranular cracking in creep. The data stimulated discussion on the mechanism by which boron affects precipitation at grain boundaries.—*M. R. Achter.*

Physical Metallurgy of Cobalt—A long research-in-progress session on the last day of such a busy week is bound to attract only the interested metallurgists. Over 50 persons were counted at one time. Abstracts for the papers presented are printed in the JOURNAL OF METALS, *10*, (20), 1958, pp 103-104.

This program ran the gamut of metallurgy, including the production of zone-refined cobalt, fundamental research on binaries, development of alloys, and statistical analyses of results obtained under various processing conditions for industrial alloys. Two papers reported properties of cobalt—hardness, tensile strength, creep, damping, and shear moduli. A series of binary cobalt alloys are being studied by the staff at Armour Research.

The precipitation phenomenon in Co-Ta alloys was investigated by the researchers of Haynes Stellite. Professor Parr discussed his findings on the behavior of the Ti_2Co phase in Ti-rich binary alloys. E. F. Adkins (Battelle Memorial Institute) reported some preliminary work on the effect of various materials dispersed in pure cobalt which was encouraging; however, the same technique used with L-605 was disappointing.

Hume-Rothery factors were used as a basis for developing binary, ternary, and other multicomponent combinations for good electrical resistivity, according to J. C. Stewart, also of Battelle.

M. J. Stutzman, Westinghouse Electric Company, presented an interesting paper on the mechanical properties and oxidation resistance of HS-31, HE-1049, Udimet 500, and Guy-alloy, melted in vacuum and in air or argon. Considerable statistical analysis was applied to reach conclusions.

Dr. L. Habraken, Chief Engineer, Centre National de Recherches Metallurgiques of Liege, Belgium, presented two papers of work sponsored by the Centre d'Information du Cobalt, Brussels, Belgium. He discussed the influence of cobalt in a chromium (12.5 pct) stainless steel (0.1 pct C). Cobalt up to 5 pct has one effect, while above this and to 12 pct shows a different effect on the final TTT diagrams.

The quaternary Fe-Cr (9-17 pct)-Co (0.10 pct)-C (0.06 and 0.2 pct) has also been studied. Cobalt does not effect the liquidus and solidus, but does broaden further the gamma phase region enlarged by chromium. This work is being continued with alloys at higher carbon contents.—*F. R. Morral.*

Physical Chemistry of Ternary Systems—This session, attended by 50 people began at 2:00 PM on Thursday, Feb. 20. In the first paper by Darken, he presented a new method of coordinating the complicated data of the ternary systems. He used activity-temperature plots which have the advantage that they can be extended into meta-stable regions. At the invariant points there is a relation between the slopes of the activity temperature curves.

Darken also showed charts for the Fe-O system, for the Fe-C system, and for the Fe-Mn-C system. In this ternary system he showed the activity of manganese as a function of temperature. Darken stated that by simultaneous solution of thermodynamic equations one can determine the change of activity along the univariant curves. He gave the mathematical, as well as the graphic solutions, and showed the close agreement with observed data. He further stated that even in those instances where we do not have good data, we can still draw some very useful conclusions.

The second paper, by Butler and McCabe, was presented by Butler, and in this paper activity data were presented as determined by the vapor pressures of manganese measured over some alloys of iron, carbon, and manganese in the temperature range of 800° to 1050°C. These measurements were made by means of a Knudsen apparatus with a vacuum micro balance. They presented a series of activity data as determined by this apparatus.—*Karl L. Fetters.*

Extractive Metallurgy Division

Refractories and Some of the Atomic Age Metals—This—one of two opening sessions of EMD—was characterized by the interest shown in the papers as evidenced by the vigorous questioning of the authors and the animated discussion. The first paper, entitled *Refractory Usage in Titanium and Zirconium Metallurgy*, prepared and presented by Alva H. Roberson, Supervising Metallurgist, USBM, Northwest Electrodevelopment Laboratory, Albany, Oregon, pointed out the refractories problems encountered in each of the successive steps in the almost identical metallurgy of these two elements and outlined present practice. The greatest difficulty is encountered in the final phase, that is, melting and casting the sponge. Mr. Roberson pointed out that, to date, only one satisfactory melting crucible is available—and that is not a conventional refractory but instead a water-cooled crucible, usually made of copper with diameters in commercial practice ranging up to 30 in. and depths of 60 in. Actually, the real refractory is a skull of solidified metal which is quite effective in minimizing contamination.

Owing to the inability of the senior author, Philip J. Clough, of the National Research Corp., Boston, Mass., to negotiate the New England climate, the second paper, with R. H. Singleton as the co-author and entitled *Usage of Refractories in Vacuum Metallurgy* was presented in abstract form only. The paper pointed out that the usual considerations of solubility and reaction between liquid metals and refractories while necessary, are not sufficient in vacuum metallurgy since additional reactions tending to generate gases or vapors will be favored under vacuum.

The third and final paper of the session was entitled *Cerium Monosulfide, a Unique Refractory* with L. M. Litz of the National Carbon Research Laboratories as author. Cerium monosulfide is one of Nature's most thermodynamically stable sulfides and therefore is quite resistant ordinarily to attack by molten

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substances; however, it is subject to oxidation at elevated temperatures and must be heated in vacuum—1900°C being about the upper limit—or inert atmospheres. It is a golden yellow metallic-appearing substance with an electrical conductivity exceeding that of mercury, making it an excellent susceptor for induction heating applications. It is fabricated into crucibles and other shapes by the common powder metallurgical techniques, such as cold-pressing, sintering, etc. The author also discussed results obtained when it is used for melting such metals as titanium, zirconium, vanadium, cerium, and copper.—*A. P. Thompson.*

Copper—Three papers were presented: 1) *Flash Smelting of Copper Concentrates* (Outokumpo Oy, Finland, by Messrs. Petri Bryk, John Ryselin, Jorma Honkasalo, and Rolf Malmstrom, presented by Mr. Malmstrom; 2) *Economics of Direct Reverberatory Smelting as Compared to Roasting and Smelting* by Royale J. Stevens of Kennecott Copper Corp., presented by L. E. Cole of American Metal Climax Inc.; and 3) *Reverberatory Smelting at Chuquicamata, Chile*, by G. E. Morris, presented by C. M. Brinckerhoff of Anaconda Co.

Mr. Malmstrom reviewed the development of flash smelting of Copper concentrates at Outokumpo, including heat balance calculations, fuel economy, SO_2 collection, and metal losses. He showed how the flash smelter combines into one unit the three phases of roasting, smelting, and converting, thus achieving considerable fuel saving. With favorable feed materials and other conditions these fuel savings may be sufficient to permit autogenous smelting.

Since Mr. Stevens' plane was delayed, his paper was presented by Mr. Cole. This paper reviewed the economics of direct smelting of wet copper concentrates as compared to the two-step operation of roasting and smelting. Advantages and disadvantages of both methods were given and trend toward direct smelting noted.

In order to handle the concentrate from the sulfide plant at Chuquicamata in Chile, a reverberatory smelting furnace was constructed and put into operation in 1952. The design, construction and operating data are reviewed in Mr. Morris' paper. In Mr. Morris' absence the paper was presented by C. M. Brinckerhoff of the Anaconda Co.—*W. L. Brytczuk.*

Rare Earths and Miscellaneous Metals—At the Monday afternoon session two papers on rare earths, one on rhenium, and one on beryllium were presented. W. G. Gruzensky and G. T. Engel, U.S.B.M., presented a new method for the separation of rare earths by which it was possible to extract as much as 65 pct of the individual rare earth elements from mixtures of the same.

D. M. Rosenbaum and I. E. Campbell, Battelle Memorial Institute, discussed the scarcity of rhenium metal and some of the exceptional properties, including its extremely high melting point.

W. W. Beaver, Brush Beryllium Co., outlined a number of methods for the production of beryllium, principally by chloride extraction. In this paper he discussed the various refinements of the technique which tend to give higher purity.

R. Didchenko, National Carbon Co., presented a paper on the preparation of anhydrous chlorides of cerium and thorium which should be most useful for future work on the preparation of the pure metals of these two elements.—*William G. Wilson.*

Titanium Session—The first paper was entitled, *The Effect of Additions on the Chlorination of Metal Oxides by Calcium Chloride*, by N. F. Neuman and A. W. Schlechten, Missouri School of Mines and Metallurgy. The paper included experimental verification of the improved yield and product purity resulting from oxide additions to chlorination systems and the use of vacuo.

A paper presented by W. E. Dunn, Jr. of E. I. DuPont de Nemours & Co., Wilmington, Del., entitled, *The High-Temperature Chlorination of Titanium Dioxide Bearing Minerals*, was concerned with chlorination rates as a function of several engineering parameters as well as two different chlorinating media, chlorine and carbon monoxide and phosgene.

The final paper in the session was presented by D. H. Baker, Jr. of the Bureau of Mines Experimental Station at Boulder City, Nev., and was entitled, *Electrorefining of Titanium from Specially Prepared Ternary Alloys*, by D. H. Baker, Jr., J. R. Nettle, and T. E. Hill, Jr. This paper represented a continuation of the Bureau's systematic work in determining the capabilities and limitations of the electrorefining process for metals such as titanium. The present work is concerned with the refining of ternary alloys of titanium containing 2½ pct of each alloying element.—*W. A. Krivsky.*

Uranium Hydrometallurgy—At the Wednesday AM session the technique of precipitating UF_4 from aqueous solution by catalytic reduction was discussed by H. G. Petrow of the AEC Raw Materials Development Laboratory. By the use of SO_2, and cupric ion as a catalyst, uranium can be reduced and a pure dense precipitate is produced. J. R. Nelli, National Lead Co. of Ohio, established the basis for current uranium concentrate specifications now used in Government procurement contracts.

The latter part of the program was concerned with a paper by W. D. Charles, National Lead Co. Inc., and N. Schiff, Vitro Rare Metals Co., regarding the effect of uranium concentrate specifications on mill operations, and a talk on the development of new oxidants for use in leaching uranium ores in a Na_2CO_3-$NaHCO_3$ system by M. A. DeSesa, National Lead Co. Inc., Winchester, Mass. Cu-NH_3 was determined to be the most efficient and economical oxidant studied during this survey.—*C. K. McArthur.*

Cobalt—Over 100 persons attended this Wednesday morning session. C. R. Whittemore of Deloro Smelting and Refining Co. was able, in the time allotted, to present the history and production of cobalt in Canada, which he illustrated with slides.

The fine points in the separation of nickel and cobalt as developed at the research laboratories of the Sherritt Gordon Mines was presented by their director, V. N. Mackiw. In the preferential reduction of nickel, there is a critical time (approximately 60 min) when the ratio of cobalt to nickel becomes a maximum of 150:1. This is utilized in producing specification cobalt powder on a semicommercial scale.

Dr. S. Mellgren and Professor J. Wulff have made use of the metallic cobalt electrodeposited at the cathodes, and reduced the cobalt oxides produced at the anodes, thus obtaining high recovery efficiencies. Some of the physical characteristics of the powders produced by electrolysis and by reduction of the oxides were also reported.

The movie, *Cobalt in Katanga* presented the operations necessary to obtain copper and cobalt from the ores in the Belgian Congo. The production of electrolytic cobalt, granules, white alloy, salts, and rondelles was shown. This film is available for showing to technical groups through the Cobalt Information Center at Battelle Memorial Institute.—*F. R. Morral*

Nickel Session—On Wednesday afternoon two papers were given and a motion picture shown to an audience of approximately 125. All three were contributions from the staff of the International Nickel Co. Ltd., and all three were interesting and very well received.

Recovery of High Purity, Sulfur, Selenium, and Cobalt in Electrorefining of Nickel, presented by L. S. Renzoni, described a new process for electrorefining of nickel, in which anodes cast directly from molten matte are employed instead of impure nickel. This novel practice eliminates several high-temperature operations necessary to produce nickel metal anodes. In addition to

solving the anode casting and electrochemical problems inherent in this unusual practice, the research work resulted in processes for the recovery of high purity elemental sulfur and a selenium-rich product, while retaining the prior features of cobalt and precious metals recovery. Separation of selenium from sulfur is accomplished by distillation in a column, 98 ft tall.

Paul Queneau presented the paper, *Production of High-Grade Iron Ore at Copper Cliff, Ontario*, which describes the production of iron oxide, low in nickel and sulfur, from nickeliferous pyrrhotite at the present rate of 365,000 tons per year. Sulfur is eliminated in fluid bed roasters operating in closed circuit with cooling boilers and cyclones. Reduction of the calcine is carried out in rotary kilns. Leaching in aerated ammoniacal ammonium carbonate removes most of the nickel, copper, and cobalt, which are recovered from the liquor. The magnetite pulp is dewatered, agglomerated into 1 in. balls, and fired on a traveling grate.

F. F. Wood of INCO exhibited the motion picture *The Refining of Nickel from Sudbury Ores*, which presented in graphic and dramatic detail the various operations of nickel production.—*C. K. Conard*

Molten Salt Session—This session was held in the afternoon of Feb. 19, 1958. About 60 persons from various institutions and companies attended. There were five papers on the agenda: two on structures and constitutions, two on kinetic properties, and one on thermodynamic properties. The paper of J. O'M. Bockris was devoted to the development of a structural model for molten salt on the basis of the known kinetic properties of fused salts, such as sound velocity, diffusivity, viscosity, and electric conductivity.

G. J. Janz made a systematic review of the various physical and chemical methods used to study the constitution of molten salts. This was followed by the paper of R. W. Laity who applied the thermodynamics of irreversible processes to the transport phenomena in molten salts and derived a modified version of the Nernst-Einstein equation which offered a better correlation between some known conductivity and diffusivity data. Results on conductivity and polarization capacitance for a number of molten salts measured at 7 to 15 kilocycles per sec were presented by D. Argyriades, which were found to obey Warburg's law. The session was concluded by the paper of L. Yang and R. G. Hudson, in which the equilibrium electrode potentials and activity coefficients of a number of metal chlorides in LiCl-KCl eutectic melt were given and the effect of the size and the charge of the ions on these data discussed.—*L. Yang*

Physical Chemistry of Sulfide Systems—

Thermodynamic data on titanium sulfide were obtained by Abendroth and Schlechten, at the School of Mines and Metallurgy, University of Missouri. This was accomplished by evaluation of the equilibria of these materials with controlled $H_2S:H_2$ ratios and a new phase Ti_2S_3 was reported.

The old problem of removal of copper from iron was clarified further, although not solved, by the study of D. L. Douglass, University of Pennsylvania, which defined conditions giving rise of the liquid miscibility gap in the Fe-Cu-S-C system.

The aqueous oxidation of pyrite was examined in a kinetic study by D. R. McKay and J. Halpern, University of British Columbia.

Finally, the complexity of the roasting process was demonstrated by two separate studies of the kinetics and mechanism of sulfide oxidation. Milton E. Wadsworth, K. L. Leiter, W. H. Porter, and J. R. Lewis of the University of Utah followed the weight changes and SO_2 evolved during the roasting of Cu_2S in oxygen and in oxygen-sulfur dioxide mixtures. These measurements were supplemented by petrographic studies which demonstrated the complexity of the process which can be retarded by entrapment of product gases, although at high temperatures SO_2 catalyzes the oxidation of Cu_2O to CuO. In examining the *Mechanism of the PbO-PbS Roast Reaction*, S. J. Merrick, C. S. Samis, and E. Peters, at the University of British Columbia, observed that the vapor transport of PbS is required to explain the reaction mechanism, but that the observed stoichiometry cannot be accounted for by PbO-PbS reactions alone.—*G. Derge*

Hydrometallurgy—This Thursday afternoon joint MBD-EMD session covered the full range of process development in the uranium and vanadium field from an operating mill back to early laboratory work.

G. T. Bator described the operating plant and minor problems at the Mines Development Inc. Resin-in-Pulp Plant at Edgemont, S. D. This features a "Merry-go-Round" which slurries to the 14 banks of RIP baskets.

R. E. Musgrove described the pilot plant results on solvent extraction and recovery of vanadium from the Climax Uranium Co. solutions to obtain a higher grade product than is now being produced at their plant. The phosphate extractant gave a 92 pct V_2O_5 product. The amine solvent gave difficulties, but produced a 97 pct V_2O_5 product.

M. E. Grimes of Eldorado described their pilot plant amine extraction of uranium from the Port Radium leach liquor. Magnesia stripping was economically desirable (because all reagents have 8 to 10 cents per lb freight cost up there) but it gave some trouble. At the full-scale plant sodium carbonate stripping is expected to be used. Although it required more pounds of reagents, it gave trouble free operation in the pilot plant.

Ray Long of Dow described three types of processes still under laboratory development: 1) Leaching high-lime ores directly with the organic solvent which saves 90 pct of the acid cost; 2) Extracting slurries which saves filtration costs, but presents an entrainment problem which is being combatted by wetting agents. 3) If the final solution from present ion exchange or solvent extraction plants is not precipitated to yellow cake, but is treated by an extra stage or two of solvent extraction, a UF$_4$ product of promising purity is obtained. This may develop so that the larger mills can bypass the refinery.—*John Dasher.*

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Slags and Slag-Metal Reactions—In an electrochemical study Dukelow and Derge found that FeO-MnO-SiO_2 melts are mixed conductors in which the current efficiency varies with silica content from under 10 to over 90 pct and with the replacement of FeO by MnO resulting in higher current efficiencies over most of the range studied. There was spirited discussion of the authors' interpretation of positive hole conduction.

Two papers dealt with the kinetics of reactions between slags and carbon-saturated iron. Kirkbride and Derge concluded that the sulfurization of iron by high-sulfur slags involves the transfer of electrons from S^{2-} to Si^{4+} or other reducible positive ion, and that diffusion of the electron-accepting species across an interface film offers a reasonable explanation of the rate controlling process under most of the conditions studied. Campbell and King observed the effect of stirring on the rate of reduction of FeO from a slag and postulated that this process is also transport controlled.

Bishop, Grant, and Chipman reported activity measurements for MnO and $FeO_{(l)}$ in complex slags of interest in open-hearth practice.—*W. O. Philbrook.*

Physical Chemistry of Liquid Iron Solution—This was a precedent-shattering session for many people. For a long time it has been believed that iron and nickel formed ideal or nearly ideal solutions in the liquid state. Speiser, Spretnak, and Jacobs on one hand and Zellars and Morris on the other both proved this concept to be untrue and showed that the deviation from ideality was con-

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siderable over wide ranges of composition. Despite the fact that the two sets of investigators used two different methods of actual and relative vapor pressure measurements, the agreement in the final activity figures was unusually close.

Floridis and Chipman presented data which showed that the activity coefficient of oxygen in pure iron is not always equal to unity but obeys the equation $\log f_o = -.20$ [% O]. Their work on the activity of oxygen in iron alloy solutions will be very welcome to people interested in high alloy steels and complex iron alloys.

Delve's short paper on the solubility and activity of titanium in carbon-saturated iron was of far-reaching interest to titanium people and to those concerned with the behavior of titanium and nitrogen in steels.

Cerium in Steelmaking—An abstract of this session held Tuesday morning at the convention is as follows: *Equilibrium Between Cerium and Sulfur in Liquid Iron* by F. C. Langenberg and J. Chipman. The product, [% Ce] [% S] was determined for iron and iron containing 17 pct Cr and 9 pct Ni. The value at 1600°C was given as 1.5×10^{-4}. The experimental heats were made in a pure CeS crucible. The stable equilibrium product was identified as CeS in the metal. It was a brassy yellow.

Determination of the Cerium Habit in Carbon Steel by R. H. Singleton. Vacuum refined steel melts containing specified amounts of sulfur were exposed to various atmospheres of oxygen and nitrogen. Cerium metal was added at a predetermined time before pouring the 20 lb heat into a mold. The product, [% Ce] [% S], varied from 1×10^{-3} to 1×10^{-4}. Metallographic and inclusion extraction techniques were used. Cerium was most frequently found to exist as an orange-red cerium sulfide. Cerium oxide tended to form a slag. Cerium nitride was not found.—*J. C. Fulton.*

Howe Memorial Lecture—A full house, standing room only, of over 450 of the foremost representatives of the industry greeted Dr. Maxwell Gensamer as he was presented for the Henry Marion Howe Memorial Lecture.

Dr. Gensamer was introduced by H. B. Emerick who cited the distinguished career in teaching and science that preceded and warranted this award. Dr. Gensamer responded with a tribute to Dr. Howe, endorsing Dr. Kinzel's earlier tribute and then analyzed and summarized a tremendous amount of reported as well as much original research in a presentation on *The Structure of Metals and the Strength of Structure.* In 1½ hr Dr. Gensamer demonstrated, as only a highly competent and learned professor can, the present state of our knowledge on why and how metals fail. The meaty and extensive charts and accompanying discussion clearly showed that, although we are still a long way from a complete understanding of the phenomena of fracture of a metal, let alone a structure, very good progress has been made and substantial usable data and trends and available to designing engineers. Dr. Gensamer received many personal congratulations on the excellence of this award paper.—*A. R. Lytle.*

Behavior of Metallurgical Systems—This well attended session heard B. C. Allen present the paper, *Surface Tension in Contact Angles in Some Liquid Metal—Solid Ceramic Systems at Elevated Temperatures* by B. C. Allen and W. D. Kingery. Surface tension measurements were made by the Sessile drop method on iron, cobalt, and nickel, and alloys of these three metals with carbon and on copper and tin. Careful purification gave values somewhat higher than those previously measured. The wetting behavior of tin and tin-titanium alloys on alumina, silicon nitride, molybdenum silicide, and silicon carbide were also investigated.

V. F. Zackay presented an interesting short paper, *Air Melting of Iron Aluminum Alloys*, showing that a careful deoxidation technique using cerium could be used to make high-aluminum alloys which could be fabricated satisfactorily and which had reasonable ductility. The method is of interest in view of the present use of other techniques, such as vacuum induction melting.

B. Trentini and M. Allard of IRSID, France were present for the presentation by Trentini of the IRSID paper, *Oxygen-Lime Injection. A New Steelmaking Process.* This paper was also illustrated by a good film. The technique of injecting powdered lime along with oxygen in a vessel similar to a conventional LD converter offered some important advantages, including very efficient dephosphorization and desulfurization, a slag which contained less iron, and improved lining life. The lance had to be raised above the levels used for oxygen injection alone because of the high momentum jet. Fumes were much less troublesome when lime was injected along with the oxygen. A lively discussion continued informally after the close of the session.—*T. B. King.*

ISD and MBD Joint Session Pyrolysis and Agglomeration—The joint session of the ISD and MBD was received by an attentive audience of about 200. *Fluosolids Roasting of Sulfides for Recovery of Copper, Iron and Sulfur*, by H. Kurushima, President of Dowa Mining Co., Japan, and R. M. Foley, Dorr-Oliver Inc., was presented by Mr. Foley who described the Dowa Mining Company's new plant for fluosolids roasting Yanahara pyrrhotites. The new plant recovers about 82 pct of the copper in the ore by acid leaching the calcine from the roaster. Furthermore, 99 pct of the iron is converted into sinter containing 60 pct Fe and less than 0.2 pct Cu.

Prior to the presentation of the remaining papers, all of which were devoted to the R-N process, Dr. Alex Stewart, President of the R-N Corp., gave a short resumé of the development of this process. Dr. H. K. Work of New York University and consultant for the National Lead Co., presented *Beneficiation of Low-Grade Iron Ores Involving Direct Reduction*, by A. Stewart and H. K. Work. He described the reduction of low grade iron ore with non-metallurgical solid fuels in a rotating kiln at temperatures between 1800° and 1900°F (below the fusion temperature of the charge). By repeated grinding of the material discharged from the kiln followed by magnetic and/or gravity concentration, the iron is separated from the gangue to yield a high-grade product containing as much as 95 pct total iron and a low-grade product containing about 80-90 pct total iron. After briquetting, the high grade product can be used in electric or open-hearth steelmaking furnaces, and the low grade can be used as blast-furnace feed.

The next paper was *Reduction of Adirondack Magnetite Concentrates in the R-N Process and Melting Tests on the Briquetted Product*, by D. E. Babcock, Research Metallurgist and E. C. Smith, Director of Metallurgy and Research, Republic Steel Corp. Dr. Babcock described the operation of the R-N pilot plant at Birmingham, Ala., where 2064 tons of briquettes containing 95.2 pct iron (92.4 pct reduced) had been manufactured from magnetite concentrates containing about 69 pct iron.

E. C. Smith then described the successful conversion of the briquettes into steel in the electric furnaces at the Republic Steel plant in Chicago using briquettes for up to 60 pct of the charge.

Dr. Herasymenko of New York University delivered a paper, *Direct Reduction of Iron Ores High in Phosphorus and Sulfur*, by A. Stewart, G. G. Reed, Jr., and P. Herasymenko which explained the mechanism for solid state transfer of phosphorus and of sulfur both from the ore and the fuel to the non-magnetic portion of the charge. Regardless of whether the phosphorus or the sulfur content of the original R-N charge was high or low, the final product would usually contain only 0.03 to 0.07 pct P and 0.02 to 0.05 pct S.

The last paper, *Recovery of Iron Ores Containing Titanium, Manganese, Chromium and Alkali Metal Sulfates*, by A. G. Oppegaard, O.

Mokleburt, and G. G. Reed, Jr., of the National Lead Co., was presented by Mr. Reed. He showed by laboratory results that a high grade product containing 90 to 95 pct iron could be obtained with the R-N process from ore containing many impurities that would make them unsuitable for blast-furnace smelting. In the R-N process 55 to 80 pct of the TiO_2, 75 to 95 pct of the MnO, 60 to 90 pct of the chromium oxide, and 97 pct of the alkaline metal sulfates are rejected into the non-magnetic tailings. —*R. L. Stephenson.*

Technology of Iron and Steelmaking Processes—Over 100 people came to hear the first public reporting of the method in use at the Duquesne works, U. S. Steel Corp. for vacuum casting of large forging-in grade ingots. J. N. Hornak presented the paper and reported that the system has consistently produced ingots with approximately 1.5 ppm hydrogen after pouring.

The results of a tracer study on the gas flow and distribution in the small experimental blast furnace at the Pittsburgh Station of the Bureau of Mines was reported by Prof. W. O. Philbrook, Carnegie Institute of Technology. It was found that the gases from a particular tuyere rose almost vertically in the shaft and did not intermingle appreciably with the gases from other tuyeres. N. B. Melcher reviewed a number of the programs of research where the U. S. Bureau of Mines' small furnace has been used and pointed out how it might be used in further studies on ore reduction and blast performance.

The meeting closed with a description by R. H. Jones of Bethlehem Steel Co. of a new open hearth model. In some cases the model duplicates flow conditions of the big unit, but this is not always the case; Consequently, results of model studies must be used with discretion in the design of full scale units.—*J. F. Elliott.*

Golden Jubilee Celebration

Commemorating its 50th anniversary, the Institute of Metals (British) held its Golden Jubilee Spring Meeting in London, from April 28 to May 2, 1958.

The weeks technical sessions and symposium were keynoted by all-day visits to various companies in the metal industry, which included the research laboratories of the British Aluminum Co. Ltd., and the Endfield Rolling Mills Ltd.

Lord Tedder, Marshall of the Royal Air Force, was inducted as the new president of the Institute at the annual general meeting.

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ERLE VICTOR DAVELER

Memorial Resolution Prepared By Andrew Fletcher

WHEREAS, With the death of Erle Victor Daveler on November 11, 1957, the Nation lost one of its renowned engineers, and the Institute a most devoted member, who had served with distinction as a Director from 1929 to 1951, as Vice-President from 1940 to 1949, and for many years as Chairman of the Investments Committee, helping to pilot the Institute through several critical years, and in 1949 became Honorary Member, and

WHEREAS, during his lifetime, he served as an officer of many mining and allied corporations, developing some of the Nation's important resources in gold, copper, lead, zinc, iron and cement, and

WHEREAS, throughout his entire career he was an inspiration to his associates by virtue of the high standard he set for himself.

THEREFORE, BE IT RESOLVED, that the American Institute of Mining, Metallurgical, and Petroleum Engineers, express its deep sorrow over the loss of this distinguished member and friend; and

BE IT FURTHER RESOLVED, that this resolution be spread upon the Minutes of this meeting and a copy be sent to Mrs. Daveler.

February 16, 1958

Third Annual Uranium Symposium at Moab

The Third Annual Uranium Symposium of the AIME Uranium Section is being staged at Moab, Utah on May 9 and 10, 1958. The meetings will be highlighted with field trips through the mill of the Uranium Reduction Co., and through the mines of the Big Indian district in San Juan County, Utah.

Max Pierson and William McDougald are serving as co-chairmen of the Geology Program; John Mullen and William Franklin as co-chairmen of the Mining Program; and officiating as co-chairmen of the Metallurgy Program, Theodore Izso and Gerald Stocks.

The Metallurgy Program will include a discussion by Harry Gardner of the National Lead Co. Inc. titled *From Ore to Green Salts.*

Topics to be covered in the Symposium are: Special problems in uranium sampling; crushing of wet ore; solvent extraction, special problem; use of chloride elution in resin –in pulp, personnel policy and procedures; precipitation factors in uranium milling; and automation in uranium milling.

1957 McFarland Award Winner

Carl Franklin Hoffman, supt. of blast furnace dept., Sparrows Point works, Bethlehem Steel Co., was the 1958 recipient of the *David Ford McFarland Award* for achievement in metallurgy. He recieved the Award at a dinner-meeting of the Penn State Chapter of the ASM on May 2, 1958.

The McFarland Award was established in 1949 by the Penn State Chapter of ASM as an annual recognition to a metallurgy alumnus of the Pennsylvania State University.

C. F. HOFFMAN

Hoffman has been active in technical committee work for AIME and AISI. He was vice-chairman of the Iron and Steel Division of AIME from 1949 to 1950, and is currently a member of that division. The *Joseph E. Johnson Award* of AIME was presented to him for his work on low-silicon pig iron, and in 1934, *Modern Industry* named him top superintendent of the year.

Hoffman has served as chairman of the AISI technical committee on blast furnace practice from 1948 to 1953.

Metal Show at Dallas

The ASM first Southwestern Metal Exposition and Congress will be held May 12 through 16, 1958 at Dallas, Texas. Emphasis will be on aircraft materials; fabrication techniques; high temperature steels and alloys; and metals problems in the petroleum and chemical industries.

Included on the Metal Show's program are 29 scientific presentations, a major paper sponsored by the Texas chapters of the Metals Treating Institute, and a four-day technical program by the Society for Non-Destructive Testing.

Small Specimens and Imperfections to be a Topic at Fall Meeting

In recent years few developments have captured the imaginations of metallurgists as much as the unusual properties of metals with very small dimensions. The superstrengths of tiny metallic whiskers, and the super-magnetic properties of thin metallic films have excite all of us. The imaginative speculations about small defects, such as vacancies in metals, many sometimes seem unwarranted. Nonethe-less, this field of science is rapidly developing and is very important to metallurgy.

The Effects of Small Dimensions (whiskers, thin films, etc.) and *Point Imperfections* (vacancies, interstitials impurities, etc.) will be the themes of groups' lectur by outstanding scientists at the Fall Meeting of The Metallurgical Society of AIME at Cleveland. These lectures are being sponsored by the Chemistry and Physics of Metals Committee, IMD. Active discussions of these lectures will be encouraged.

Also at the Fall Meeting of The Metallurgical Society of AIME, the Physical Metallurgy Committee of IMD is planning a symposium on the *Effect of Ordering on Mechanical Properties.* The symposium will include invited papers on this topic, dealing with several different ordering systems. The committee is interested in other papers of an engineering nature, giving examples of where ordering is an important consideration in practical problems.

Abstracts should be 200 to 300 words in length (no photographs, diagrams, or tables can be used.) Three copies of each abstract should be sent to: *IMD Programs Committee, Metallurgical Society of AIME, 29 West 39th St., New York 18, N. Y.*

The deadline for submission of abstracts is July 15, 1958.

If the author wishes his paper to be published, he should submit three copies of the manuscript to the IMD Publication Committee. Selection of an abstract for presentation at the symposium does not necessarily assure publication of the manuscript in the JOURNAL OF METALS, nor does approval for publication of the manuscript assure presentation of the paper at the Physical Metallurgy Symposium.

News of EDUCATION

► **The Massachusetts Institute of Technology** will offer a Special Summer Program in the *Casting of Light Metals* during the 1958 Summer Session, from June 17 through June 21, 1958.

This Special Summer Program will deal with casting alloys of aluminum, magnesium, and titanium and with the important applicable foundry processes, including sand, plaster, permanent mold, and die casting.

The Program will meet daily, with Sessions both mornings and afternoons, during the week. It will include lectures, and laboratory demonstrations, with approximately equal time given to theory and practice.

Tuition for this one week program will be $200, due and payable upon notification of admission. Academic credit will not be offered.

MIT is also offering a two-week Special Summer Program in the *Metallurgical Applications of X-ray Diffraction*, from August 18 through August 29, 1958.

The program will have lectures in the mornings and laboratory demonstrations and discussions in the afternoons. Topics to be considered include: emission and absorption of X-rays; the diffraction process; interpretation of powder diffraction patterns; precise lattice constant determination; application to study phase diagrams; film cameras and diffractometers; crystal orientation; texture and preferred orientation; measurement of residual stress; and X-ray flourescent analysis.

Lecture material is planned especially for the metallurgist who has not had advanced work or who may have been out of school for several years. However, an elementary knowledge the nature of X-rays and the structure of metals is necessary. The program is not a research conference, and is stressed to teach the subjects, rather than to present a review.

Tuition for this two-week program will be $250, due and payable upon notification of admission. Academic credit will not be offered. Exercises will be held from 9:30 to 12:30 pm and 2:00 to 5:00 pm, Mondays through Fridays.

► College juniors of exceptionally good records in engineering and science are invited by **Rensselaer Polytechnic Institute,** Troy, N. Y. to apply for a scholarship in the Fourth Annual Summer Conference for Advanced Undergraduate Students, July 21 to Aug. 30, 1958. Two concurrent courses will deal with the physical chemistry of metals and modern metal physics. The 20 scholarships to be awarded will cover tuition, living expense, and travel costs.

Each of the courses in the program will provide 45 hr of lecture or its equivalent, the student earning 3 hours of credit for satisfactory completion of each. The courses are acceptable for senior year or first year graduate credit at Rensselaer and thus may be acceptable as part of the degree program at the institution currently attended by the student. Should the student later be admitted for graduate work at Rensselaer, the credits earned in the summer conference may be used towards a master's degree.

► The 1957-59 edition of the *ECRC Engineering College Research Review*, 408 pages, is now avalible for distribution at $2.00 per copy. It provides an exhaustive analysis and location guide for engineering and associated science activities and capabilities in colleges and universities throughout the country. A full and carefully-compiled subject index of more than 50 pages is supplied. The *Review* is edited by Renato Contini of New York University. Copies, order blanks, and further information may be obtained from *Renato Contini, ECRC Secretary, University Heights, New York, 53, N. Y.*

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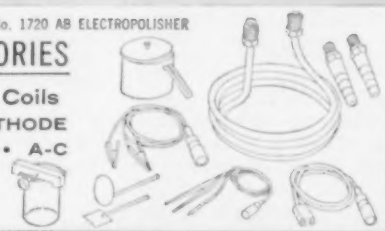

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AROUND THE SECTIONS

Cleveland Section Holds One Day Symposium

One-hundred forty registrants gathered at the Hotel Pick-Carter in Cleveland on March 20 for a one day symposium on metal forming and machining.

The morning session was devoted to the general aspects of forming, machining, and forging. G. M. Cover, of the Case Institute of Technology, Cleveland, and A. M. Montgomery, of Alcoa, Cleveland, were chairmen.

After lunch at the Lorenzo Carter Room, three separate sessions were held. These sessions concentrated on the subjects: *Fabrication of Light Sections*, with co-chairmen W. W. Beaver, Brush Beryllium Co., Cleveland, and N. P. Goss, Consultant, Cleveland; *Machining*, under J. R. Kennedy, Cleveland Twist Drill Co., and R. H. Akers, Chandler Products Corp., Cleveland. *Forging* was the subject of the third session at which A. Grobe, Thompson Products Inc., Cleveland, and H. Maring, Alcoa, Cleveland, acted as chairmen.

Following the afternoon sessions, a dinner was held in the Ballroom at which R. E. Rickesecker, Chairman of the Cleveland Section acted as toastmaster. Speaker of the evening was H. Y. Hunsicker, asst. chief, Cleveland Research Div., of Alcoa, who discussed *Fourth Dimensional Photography*, otherwise known as "time lapse".

W. M. Baldwin, Jr., served as chairman of the Symposium Committee, and working with him were A. Anderson, A. S. Nemy, and E. P. Weber.

Several Indian Engineers participating in the *INSTEP* program took part in the Symposium.

• The seventh meeting of the **Boston Section** was held on Monday, April 21st in the Experimental Foundry, of the Massachusetts Institute of Technology, Cambridge, Mass.

Albert G. H. Dietz, Prof. of Building Engineering at MIT, was the featured speaker. Dietz, an MIT graduate, is well known for his work on plastics as materials of construction. His talk, entitled, *Engineering Plastics*, showed what plastics are and how their particular properties are derived from their basic structure. He also discussed high strength plastics, with fibrous reinforcement, which provide a means of tailoring materials with a high strength:weight ratio.

The Student Metallurgical Society of MIT, which draws its members from students who belong to AIME, ASM, and AFS, acted as hosts and were entirely responsible for the program and the arrangements.

• Tours of the facilities of the University of Illinois College of Engineering, and the Illinois Geological Survey, at Urbana, Ill. keynoted the start of the April 11th meeting of the **St. Louis Section.** Later, an evening dinner was held in the Garden Room of the Hotel Urbana-Lincoln, with Hubert E. Risser, mineral economist, Illinois Geological Survey, as the guest speaker.

His talk, *Metallurgical Coke From Illinois Coal*, briefly described some of the research and development in the use of Illinois coal for the production of metallurgical coke. The discussion covered the declining reserves of high-rank coking coal, and the resulting need for adaption of Illinois coals and others to replace those now most commonly used.

• The **Philadelphia Section** honored the United States Steel Corp., for its accomplishment in the construction of the Fairless works, at a dinner-meeting held in the Hotel Hildebrecht, Trenton, on March 20.

U. S. Steel Night at AIME, was the theme for the evening, which featured two prominent U. S. Steel engineers as guest speakers. Ralph C. Beerbower, general superintendent, Frick district coal operations, Uniontown, discussed new developments in mechanized coal mining, including the latest equipment for removing bituminous coal from the underground seam, under-ground transport, and on-ground handling.

Eugene L. Bartolotta, chief steel production metallurgist at the Fairless works, was the other guest speaker. He talked on sulfur control in open-hearth steelmaking, the sources of sulfur in molten steel, the removal of sulfur through good slag practice, and the effects of improper control on steel quality.

Research Center Welcomes Physical Metallurgy Group

On Monday, Mar. 24, 1958, the American Brake Shoe Co. played host to the Physical Metallurgy Group of the New York Section of AIME, at the company's Research Center in Mahwah, N. J. The Research Center carries on research, and development work for the divisions of American Brake Shoe Co.

Three principal laboratories of the center, the metallurgical, chemical, and mechanical, were visited by the group. Joseph J. Connelly, research metallurgist of American Brake Shoe Co., welcomed the group to the laboratories. In an orientation briefing, Connelly summarized work of the American Brake Shoe Co. in serving the railroad, automotive, aircraft, mining, and construction industries.

At the metallurgical laboratory, headed by F. B. Herlihy, the visitors were shown facilities devoted to metallography, studies of creep, and the experimental foundry with equipment for testing car wheels, preparing high-purity aluminum castings, and analyzing for defects in castings by radiographic and X-ray techniques.

The Chemical Laboratory, headed by Frank Smith, is concerned with fundamental research on synthetic resins, which have applications in brake shoe linings and in anti-friction materials. Development, testing, and evaluation of asbestos brake linings is carried on in this department. Research is also being carried out on powdered metal friction materials.

The Sargent Research Laboratory, of which H. H. Harris is in charge, is devoted to mechanical research. It is the center for research on railroad bearings, wheel brakes and dynameter testing equipment.

After the plant tour, the Physical Metallurgy Group were guests of their hosts for dinner at the Otis-Cutler Club in nearby Suffern, N. Y. In a technical meeting that followed, Arthur S. Nowick of the Watson Laboratory of International Business Machines spoke on *International Friction in Alloys*, describing the relation between internal friction and temperature. At high temperatures atoms can interchange positions so rapidly that internal friction is low, while at low temperatures, atoms change positions so slowly that internal friction is again low. It is the intermediate range in temperature that is interesting to study. Dr. Nowick's studies were based on a study of 30 pct zinc-silver and 4 pct copper-aluminum alloys.

The technical chairman, Howard Avery of American Brake Shoe Co., introduced Dr. Nowick. Presiding at the meeting was Alfred Bornemann, chairman of the New York Physical Metallurgy Group.

Gold Medal Winner

Col. Donald M. Liddell was recently awarded the Mining and Metallurgical Society of America's *Gold Medal* for "unselfish contribution in the art of nonferrous metallurgy and research in connection therewith." The presentation of the Medal was made at the Society's annual dinner on Jan. 29, 1958, at the Princeton Club in New York City.

Liddell, who is also a member of the Metallurgical Society of AIME, was the first metallurgist to produce selenium and tellurium on a tonnage basis. He did this in 1905 and 1906, only a few years after graduating from John Hopkins University. The author of three important books: *Metallurgists, and Chemists' Handbook; Handbook of Non-Ferrous Metallurgy; and Principles of Metallurgy.* He has also held positions as associate and managing editor of the *Engineering and Mining Journal.*

Col. Liddell served as secretary of the Mining and Metallurgical Society of America for seven years, and also officiated as president of the Society for one year.

New Cobalt Reference Lists

The Cobalt Information Center, Battelle Memorial Institute, has announced the availability of three new reference lists on Cobalt, as listed below.

Aluminum-Cobalt Alloys, a four page bibliography contains references to literature published on cobalt in aluminum alloys from 1908 to mid-1957. Also listed are United States and Foreign patents from 1897.

Cobalt in Cast Iron is a one page list of literature references and patents from 1930 to 1956.

Cobalt in Stainless-Steels contains one page of references to literature and patents from 1941 to 1957. Included are heat resistant and high-creep-strength austenitic steels and alloys.

These publications may be obtained by addressing requests on company letterhead to the *Cobalt Information Center, c/o Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio.*

Reactor Materials

A lecture course for practicing metallurgists on *Materials for Nuclear Reactors*, will be held at New York University's Bronx campus from June 9 through June 13. Sponsored by the NYU College of Engineering, the course will include 20 subjects on these general topics: criteria for materials selection, properties for materials for reactor applications, environmental (including radiation) effects, fuel materials and systems, and general problems in reactor component design.

Other specific topics to be covered are: the metallurgy of thorium and plutonium, ceramics and graphite, zirconium and beryllium, radiation in ceramics and cermets, uranium base fuel systems, ceramic fuel systems, heat transfer design considerations, and trends in reactor development.

Chairman of the course is Dr. Irving Cadoff, assoc. professor, NYU Dept. of Metallurgical Engineering.

MEMBERSHIP

Proposed for Membership
Metallurgical Society of AIME

Total AIME membership on Mar. 1, 1958 was 33,267; in addition 3,113 Student members were enrolled.

ADMISSIONS COMMITTEE

J. H. Scaff, Chairman; Alfred Bornemann; F. B. Foley; T. D. Jones; L. L. Seigle; J. M. Warde; H. K. Worke.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

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REINSTATEMENTS

To Member

Carlos J. Antezana, Catavi, Boliva, S. A.

To Associate

Oscar T. Ward, Alamogordo, N. M.

REINSTATEMENTS—CHANGE OF STATUS

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Clayton O. Matthews, Palo Alto, Calif.
Gordon W. Powell, Madison, Wis.
William H. Smith, Schenectady, N. Y.

Student to Junior

Sid W. Turner, New York, N. Y.

NECROLOGY

Date Elected	Name	Date of Death
1916	L. M. Allen, Jr.	Feb. 12, 1957
1923	Walter S. Bourlier	Dec. 1957
1955	Jack E. Chiesler	Nov. 1957
1947	Edward J. Fearing	Feb. 18, 1958
1919	Wilbur H. Grant	Dec. 28, 1957
1953	Sturgis W. Macomber	Aug. 1, 1957
1918	Fred H. Moffit	Unknown
1914	Charles R. Stahl	Dec. 24, 1957
1909	Clinton M. Young	Oct. 14, 1957

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COMING EVENTS

May 9-11, AIME Uranium Section, Third Annual Uranium Symposium, Moab, Utah.

May 12-16, Southwestern Metal Exposition and Congress, State Fair Park, Dallas.

May 21, NOHC Detroit Section, Hotel Fort Shelby, Detroit.

May 27-29, AIME Third Reactive Metals Conference; host section: Niagara Frontier Section; Statler Hotel, Buffalo.

June 22-29, American Society for Testing Materials, Annual Meeting and Exhibit, Hotel Statler, Boston.

Oct. 3, 4, AIME NOHC Southern Ohio Section, fall meeting, Deshler-Hilton Hotel, Columbus, Ohio.

Oct. 16, AIME Utah Section, *Progress in Air Pollution Control*, Salt Lake City.

Oct. 27-30, AIME Fall Meeting of The Metallurgical Society, sponsored by the Institute of Metals Division, Carter Hotel, Cleveland.

Nov. 17-20, The Fourth Conference on Magnetism and Magnetic Materials, sponsored by the American Inst. of Electric Engineers, in cooperation with The American Physical Soc., the Inst. of Radio Engineers, The Metallurgical Soc. of AIME, and the Office of Naval Research, Sheraton-Hotel, Philadelphia.

Dec. 3-5, AIME 16th Electric Furnace Conference, Statler Hotel, Detroit.

Dec. 11, AIME Utah Section, Panel discussion on the Place of Research in the Minerals Industries, Salt Lake City.

Feb. 15-19, 1959, AIME Annual Meeting, St. Francis Hotel, Sheraton-Palace Hotel, and Sir Francis Drake Hotel, San Francisco.

Apr. 2-3, 1959, AIME Technical Conference on Stress Corrosion, Mellon Institute, Pittsburgh.

Apr. 6-8, 1959, AIME 42nd National Open Hearth Steel Conference and Blast Furnace, Coke Oven, and Raw Materials Conference, Jefferson Hotel, St. Louis.

Apr. 27-30, 1959, AIME International Symposium on the Physical Chemistry of Processed Metallurgy, Penn-Sheraton Hotel, Pittsburgh.

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DATA ON VOLMEX 81 . . .

HOW VOLMEX. 81

performs as a high volatile ingot mold coating when applied . . .

(1) Wet (2) Dry (3) At varying ingot mold temperatures

VOLMEX 81 is a highly volatile ingot mold coating which has been tested and adopted by a number of steel plants. The charts below are based on actual data obtained during such trial runs. Because of the differing practices employed by various companies, we have included several variables. First, because VOLMEX 81 can be applied either dry or with water as the carrier (spraying, swabbing or dipping are all practical), we have presented test figures on its use, both wet and dry. Second, we have included data on a range of ingot mold temperatures from 100°F. to 700°F., and third, we offer for comparison the results of a one-hour and a three-hour time-at-mold temperatures. No matter what method of application was used, VOLMEX 81 was found to repel metal splashes effectively before they could solidify and cling to the mold wall. At the same time, enough of the graphite-base coating was left at the metal/mold interface to keep the main body of the pour from clinging when it reached the point of splash, thus reducing surface defects. For wet application, VOLMEX 81 is easily stirred into suspension and remains in that state for hours without re-agitation.

VOLMEX 81 is the most volatile of the United States Graphite Company ingot mold washes. Others, in order of their volatility are: 90-B (non-volatile), MEX (very little volatility), and VOLMEX (volatile, but less so than VOLMEX 81). Write us for more extensive details or our engineering staff is available for on-the-spot assistance. Mold spray and mixing equipment are ready for your use in tests you may want to arrange.

Table I **Volatility of VOLMEX 81 Ingot Mold Wash**

Mold Coating Volatility Test—VOLMEX 81 Water Application 2.75 lbs. powder/gal. water—Period—1 Hour at	Temperature 300°F.	500°F.	700°F.
% loss after heating for 1 hour	74.3	73.9	75.4
% loss on ignition at 1900°F. after 1 hour at specified temperature	16.1	14.4	11.3
Total percent volatile matter	90.4	88.3	86.7

Table II

Mold Coating Volatility Test—VOLMEX 81 Dry Application—Period—1 Hour at	Temperature 100°F.	300°F.	500°F.	700°F.
% loss after heating for 1 hour	0.1	0.8	0.9	2.8
% loss on ignition at 1900° F. after 1 hour at specified temperature	60.8	60.4	59.7	56.5
Total percent volatile matter	60.9	61.2	60.6	59.3

Table III

Mold Coating Volatility Test—VOLMEX 81 Dry Application—Period—3 Hours at	Temperature 100°F.	300°F.	500°F.	700°F.
% loss after heating for 3 hours	0.1	0.8	1.1	4.5
% loss on ignition at 1900°F. after 3 hours at specified temperature	60.6	59.6	59.7	53.1
Total percent volatile matter	60.7	60.4	60.8	57.6

You will note in the first chart which is the wet application, that the loss is all of the water contained plus a slight amount of the remaining volatile. Since on a weight basis, the powder is about ¼ of the make-up, if you will multiply the percent loss figures at 1900°F. by four, you will get an idea of the amount of volatile which would be present for loss during the pouring operation.

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